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THE EFFECT OF DIFFERENT TEST METHODS ON DURABILITY CLASSIFICATION OF MODIFIED WOOD

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Abstract:
In order to encourage increased use of wood more empirical data on the performance of wood products are needed from different exposure situations and geographical locations. In the current study modified Scots pine sapwood materials aimed treated for above ground use were compared using: 1) two different laboratory decay tests at three different climates, 2) four different laboratory moisture tests, and 3) two different field trials. The aim of the study was to investigate the effect of temperature and moisture on modified wood material performance in laboratory decay trials and to compare different durability classification methods. Lowering the temperature did slow down the decay rate, but did not make much difference in the durability ranking of the materials. The moisture behaviour of the materials in this test could not alone explain the decay resistance. The durability classification varied between the tests, confirming that the durability classification of a material, and the ranking between materials, is not a fixed value that can be based on one single test. In order to predict service life in future studies the authors recommend to combine decay and moisture data.

Key words: *fungal decay; material resistance; moisture performance; test comparison; wood modification.*

INTRODUCTION

There is an increased awareness of the beneficial properties wood as a building material can provide. But planners, including architects, developers and engineers, require documentation of reliable service life data. This causes some challenges, mainly because there is no universal service life for a given species or treatment, the performance also relies on the climatic conditions and the deteriorating organisms at a given location. If a mistake is made during construction, leading to a moisture trap, the service life of a component could drastically be reduced compared to the service life expected from a well-designed and well-built construction.

It is important to keep in mind that durability is not equal to service life. Durability is a property that provides a given service life. ''The durability of wood is its resistance to wood-destroying organisms under the influence of its environment'' (Brischke *et al.* 2006). Wood species and potential wood treatment is, obviously, of great importance for the durability. In addition external factors will influence the durability, and Brischke *et al.* (2006) listed the following factors as important: 1) material temperature and wood moisture content, 2) design and execution of details, 3) conditions of use, 4) dimensions of a component, 5) cracks, and 6) the quality of workmanship. Service life is a given time period usually specified in years. The service life ends when a property reaches its critical limit of performance. Increased service life of a wood product implies less work related to replacement and lower cost for the user. It also gives a positive environmental contribution because the longer a wood product stays in use the longer it will store its carbon, and the longer one has to wait to replace a product the better since every replacement results in an environmental impact.

Unfortunately, service life prediction of wood products has so far mainly been based on expert judgement rather than empirical studies (Brischke *et al.* 2012). Brischke and Thelandersson (2014) reviewed existing modelling approaches for outdoor performance of wood products. The first comprehensive approaches include: MacKenzie *et al.* (2007), Thelandersson *et al.* (2011), Isaksson *et al*. (2014) and Meyer-Veltrup *et al.* (2017). Apart from a few comprehensive approaches using long-term field test data, service life prediction has been attempted in the past using laboratory test results. It is still controversially discussed if and to what extent durability tests under defined laboratory conditions can be used to predict the outdoor performance of wood and the effect of different environmental parameters such as different climate variables.

OBJECTIVE

The main objectives of the current study were to: 1) investigate the effect of temperature and moisture on modified wood material performance in laboratory decay trials, and 2) to compare different durability classification methods.

MATERIAL, METHOD, EQUIPMENT

Wood materials

Scots pine sapwood from the same raw material batch was used for all treatments and control specimens (SpS). This study aimed for above ground use (use class 3) treatment levels. Samples with dimensions according to EN 252 (2.5×5.0×50 (ax.) cm^3) (CEN 2015) were treated and used directly in the field trials. For the laboratory trials subsamples were provided from EN 252 stakes using the outer part of the samples, but not including the outermost 2mm (in order to avoid any potential surface artefacts). The treatments included:

- Acetylation (Ac), weight percent gain 21%, by Titanwood, The Netherlands.
- Thermal modification (TM), Thermo D, 212°C, by Scandinavian Fine Wood, Sweden.
- Furfurylation (FA), Nordic Wood Preservation Council (NWPC) class AB, by Kebony ASA, Norway. FA treatment was performed twice in order to provide full penetration.
- Dimethylol dihydroxyethyleneurea, DMDHEU (D), 1.3M, University of Goettingen, Germany.

Reference treatments included:

- Copper HDO (Cu), NWPC class AB, commercial product and treated at NIBIO.
- Copper Chromium Arsenate (CCA), NWPC class AB, commercial product and treated at NIBIO.

Laboratory decay tests

Mini block: The sample size was according to Bravery (1979), 5×10×30 (ax.) mm^3 and the test fungus was the brown rot fungus *Postia (Rhodonia) placenta* (Fr.) M.J. Larsen & Lombard (strain FPRL 280). The tests were performed under three different climate conditions: 22°C/70% RH, 16°C/70% RH, or 11°C/70% RH. All materials were leached according to EN 84 (CEN 1997) prior to fungal exposure. A sterile soil medium was used containing 1/3 sandy soil and 2/3 ecological compost soil. The moisture content of the soil was adjusted to 95% of its water holding capacity according to ENV 807 (CEN 2001) and 20 g sterile soil was used in each Petri dish. As inoculum a liquid culture containing 4% malt was used and 1 mL was applied on each sample. The exposure time was 20 weeks and the tests were run with n = 10 replicates. The long incubation time of this non-standard test was chosen in order to provide mass losses high enough to differentiate between materials known to be durable. The weight of the plates was recorded at the start of the test and every third week. Sterile water was added when needed in order to keep the soil moisture content stable throughout the test.

ENV 807: The test was performed according to ENV 807 (CEN 2001) using specimens of 5×10×100 (ax.) mm^3 and n = 10 replicates. All materials were leached according to EN 84 (CEN 1997) prior to fungal exposure. Deviation from the standard was that all samples were harvested already after 13 weeks and that mass loss was used directly (not recalculated according to chapter A.7 in ENV 807). Three different climates were used: 25°C/85% RH, 25°C/65% RH or 10°C/85% RH.

Field decay tests

EN 252: Ten replicate stakes (25×50×500 (ax.) mm^3) from each wood type in accordance with EN 252 (CEN 2015), were prepared. The specimens were exposed from 2010 in the NIBIO test site in Ås (the test is still running) using n = 9 replicates. The stakes were assessed annually using a pick test according to the respective standard as follows: 0 = no attack, 1 = slight attack, 2 = moderate attack, 3 = severe attack, 4 = failure.

Horizontal double layer (HDL): Ten replicate stakes (25×50×500 (ax.) mm³) from each wood type were exposed in 2009 at the NIBIO test field in Ås (the test is still running) using n = 9 replicates. The specimens were placed horizontally in double layers according to Augusta (2007) with the upper layer displaced laterally by 25mm to the lower layer. Supports were 25cm above ground and made from aluminium L-profiles. The stakes were assessed annually using a pick test based on the five-step EN 252 (CEN 2015) rating scheme.

Moisture tests

Ten replicate specimens of 5×10×100 (ax.) mm³ were used for three different 24 hour tests (W24) and a capillary water uptake test.

Desorption ($W24_{0\%RH}$): Specimens were stored in 100% RH until constant mass (approx. 2 weeks) and weighed to the nearest 0.001g to determine mass at fibre saturation. The specimens were exposed directly on freshly activated silica gel and weighed again after 24h. The water release of the specimens during 24h was determined and expressed as percentage of mass at fibre saturation.

Liquid water uptake by submersion ($W24_{submersion}$): Specimens were oven-dried at 103°C until constant mass and weighed to the nearest 0.001g to determine oven-dry mass. Specimens were submersed in a container filled with demineralised water and placed in normal climate. Specimens were separated from each other by thin spacers (cross section 1×1mm²). The specimens were weighed again after 24h submersion. The water uptake of the specimens was determined and the resulting moisture content after submersion was calculated.

Water vapour uptake in water saturated atmosphere ($W24_{100\%RH}$): Specimens were oven-dried at 103°C until constant mass and weighed to the nearest 0.001g to determine oven-dry mass. The bottom of a miniature climate chamber (plastic container with stainless steel trays and ventilator) was filled with demineralised water. Specimens were exposed using thin spacers (cross section 1×1mm²) above water in the well ventilated miniature climate chamber and weighed again after 24h. The water uptake of the specimens was determined and the resulting moisture content after 24h was calculated.

Capillary water uptake (CWU): Short term water absorption was measured according to modified EN 1609 (CEN 1997) procedure using a Krüss Processor Tensiometer K100MK2. Specimens of 5×10×100 (ax.) mm³ were placed in 20°C/65% RH till constant mass. The axial specimen surfaces were positioned to be in contact with water and fixed in the Tensiometer. The specimens were subsequently weighed to the nearest 0.0001g continuously every 2s for 200s. The capillary water uptake was determined over time in g/cm².

RESULTS AND DISCUSSION

Laboratory decay tests

Mini block: Table 1 (first three columns) shows mass losses expressed as both mean (ML_{MV}) and median (ML_{MD}). Tukey-Kramer (T-K) comparisons of ML_{MV} are provided between each material within each climate. Materials not connected by the same letter in the T-K comparison were significantly different. Among the wood modifications acetylation performed best in all climates, thermal modification, furfurylation and DMDHEU performed at statistically similar levels except for 22°C where DMDHEU had a significantly lower ML_{MV} than thermal modification. CCA had a significantly better performance than the copper HDO. It has been shown that the percentage of copper and other cobiocides removed during leaching tests was higher from wood treated with alternative copper-based preservatives than that of CCA treated wood (e.g. Temiz *et al.* 2006). This can not fully explain the high mass loss of copper HDO treated wood in the present study. The poor performance of the copper HDO treated wood can potentially be due to: 1) The copper tolerant behavior of *P. placenta*. Cu-tolerance of brown rot fungi is believed to be linked to oxalic acid excretion, particularly in the initial phases of wood colonization (Clausen and Green, 2003). Oxalic acid reacts with copper in wood to form insoluble copper oxalate that is less toxic to the fungi (e.g. Humar *et al.* 2004). 2) The small sample size of the tested wood specimens. This could potentially have resulted in a very high removal of co-biocides during the EN 84 leaching procedure prior to fungal testing, predominantely boron. 3) Long exposure time (20 weeks) can have increased the effect 1 and 2 (above). One could assume that in this mini block test the control samples probably reached maximum mass loss before 20 weeks. Hence, the difference between the treated samples and control is probably underestimated.

Durability class (DC) categories based on both ML_{MV} and ML_{MD} are indicated based on CEN/TS 15083-1 (CEN 2005a). No difference was found in durability classification between ML_{MV} and ML_{MD} or between 22°C and 16°C. Table 1, left columns, provides T-K comparison of ML_{MV} between climates within each treatment. All treatments showed significant differences between 22°C and 11°C. Only thermal modification showed significant differences between all three temperatures. A change in temperature did not result in much change of the durability ranking between treatments.

Table 1

Mini block samples exposed 20 weeks to P. placenta at three different climates. Durability class categories based on CEN/TS 15083-1 (CEN 2005a); >30% DC5, >15% to ≤30% DC4, >10% to ≤15% DC3, <5 to ≤10% DC2, ≤5 DC1

	22°C/70% RH			16°C/70% RH			11°C/70% RH			Climate		
	T-K	ML_{MV} [%]	ML_{MD} [%]	T-K	ML_{MV} [%]	ML_{MD} [%]	T-K	ML_{MV} [%]	ML_{MD} [%]	22°C 70RH	16°C 70RH	11°C 70RH
SpS	A	65.5	65.7	A	65.0	65.4	A	53.4	53.2	A	A	B
Cu	A	63.3	65.6	A	53.6	57.0	B	26.3	27.4	A	A	B
TM	B	41.8	40.3	B	32.7	32.0	CD	13.7	13.5	A	B	C
FA	BC	32.4	31.4	B	31.7	31.3	BC	18.4	22.5	A	A	B
D	C	29.0	29.3	B	23.1	18.6	CDE	9.6	4.6	A	AB	B
Ac	D	1.4	1.2	C	0.1	0.0	E	0.0	0.0	A	B	B
CCA	D	0.0	0.0	C	0.2	0.0	DE	1.4	0.1	B	AB	A

ENV 807 (CEN 2001): Table 2 (first three columns) provides mass losses expressed as both ML_{MV} and ML_{MD} and T-K comparison of ML_{MV} between each material within each climate. Wood modifications had less than 3% mass loss in all climates. Mass loss categories are indicated based on CEN/TS 15083-1 (CEN 2005a). No difference was found in DC classification for the modified wood materials in this test. With longer exposure time the differences in material performances, i.e. mass loss, would have been clearer. Table 2, left columns, provides T-K comparison of ML_{MV} between climates within each treatment. For the materials with mass loss above 3% (Scots pine sapwood, copper HDO and CCA) a significant difference was found between 25°C and 10°C. It is important to keep in mind that due to the low mass loss of the treated materials in this test the comparisons between materials and climates are only indicative. In an ENV 807 test by Westin and Alfredsen (2007) modified wood performed better than both copper chromium and CCA preservatives in compost soil after 40 weeks incubation.

Table 2

ENV 807 samples exposed 13 weeks at three different climates. Durability class categories based on CEN/TS 15083-1 (CEN 2005a); >10% to ≤15% DC3, <5 to ≤10% DC2, ≤5 DC1

	25°C/85% RH			25°C/65% RH			10°C/85% RH			Climate		
	T-K	ML_{MV} [%]	ML_{MD} [%]	T-K	ML_{MV} [%]	ML_{MD} [%]	T-K	ML_{MV} [%]	ML_{MD} [%]	25°C 85RH	25°C 65RH	10°C 85RH
SpS	A	12.0	12.7	A	9.2	9.5	A	2.7	2.2	A	B	C
Cu	B	4.6	4.3	BC	1.7	0.6	BC	1.3	1.3	A	B	B
CCA	BC	3.1	2.9	BC	1.2	0.8	B	1.4	1.4	A	B	B
FA	CD	2.6	2.4	B	2.6	2.6	AB	1.8	1.9	A	A	B
D	DE	1.1	1.3	BC	1.0	1.0	B	1.4	1.2	A	A	A
TM	E	0.1	0.0	C	0.0	0.0	CD	0.4	0.3	B	B	A
Ac	E	0.0	0.0	C	0.0	0.0	D	0.0	0.0	A	A	A

When comparing the modified wood results from the sterile mini blocks exposed to *P. placenta* with the unsterile ENV 807 samples exposed to a mixed community of soil inhabiting fungi the most striking difference was for thermally modified wood. In the mini block test the highest mass loss among the wood modifications was found for thermal modification, and there were no significant differences between thermal modification and furfurylated wood. In the ENV 807 test only initial signs of decay were detected in thermally modified wood and no significant difference was found between the performance of thermally modified and acetylated wood. The explanation might simply be the low mass losses, or there might be an effect due to different wood degrading organisms.

The predictive potential will depend on the incubation time. To find the right incubation time is tricky and depends on the research question. One apprach is to always use a standarised decay test with a fixed incubation time. But Round Robin tests show that even within the boundries of a standarised test the results

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can vary greatly between laboratories. Another approach is to harvest samples at different incubation intervals, this will give a better insight regarding the dynamic of the decay in different materials, e.g. a potential lag phase or sudden acceleration of decay. However, this procedure requires a higher number of test samples.

Field decay tests

EN 252: The data for soil contact exposure in Table 3 show that Scots pine sapwood had the most severe decay and acetylated wood the least severe decay after six years of exposure. No significant difference in performance was found between the reminding treatments. The EN 252 (CEN 2015) standard states that: ''*The test shall be run for a minimum period of five years (or until all stakes have failed if this occurs earlier). It is advisable to continue the test beyond five years, with inspections at suitable intervals, in order to determine longer performance of the treated stakes. NOTE: Ideally the test should be continued until all stakes of the product under test have failed*.'' The latter is the only way to determine the actual average service life of the tested materials in the respective test.

HDL: Table 3 also illustrates that Scots pine sapwood had the most severe decay after seven years of exposure in HDL. The treated samples fell into two groups based on the T-K comparison: 1) DMDHEU, thermal modification and copper HDO with a mean decay rating between 1.6 and 2.1 after seven years and 2) furfurylation, acetylation and CCA with only initial signs of decay, mean decay rating of 0.2 or below. The HDL is believed to be comparable to worst case scenario for wood in service above ground, i.e. a badly constructed detail with a moisture trap. The HDL is a non-standard test and no guidance is given with respect to how long the test should run. To keep samples in the field trial until all stakes fail in order to provide actual service life data is a good recommendation also for the HDL test.

The preliminary durability classification based on the CEN/TS 15083-2 (CEN 2005b) approach are provided for both field trials for mean and median decay rating instead of mass loss. This is stretching the original methodology since: 1) it is based on decay rating not mass loss, 2) not all control samples have failed, 3) the four decay rating categories are broad and the evaluation method not very sensitive plus that it not nesessarily is a good linear relation between the four decay rating categories. Hence, this table is a rough way to compare the preliminary data from field trials and the DC classification is expected to change after longer field exposure time.

Table 3

Mean(MV) and median (MD) decay rating of treated wood in above ground horizontal double layers (HDL) after seven years and EN 252 (CEN 2015) after six years. The durability classes are indicated according to CEN/TS 15083-2 (CEN 2005b) based on both mean and median values: >0.80 DC5, >0.45 to ≤0.80 DC4, >0.20 to ≤0.45 DC3, >0.10 to ≤0.20% DC2, ≤0.10 DC1.

	EN 252					HDL				
	T-K	**Decay rating$_{MV}$**	**x-value**	**Decay rating$_{MD}$**	**x-value**	**T-K**	**Decay rating$_{MV}$**	**x-value**	**Decay rating$_{MD}$**	**x-value**
SpS	A	3.0	-	4.0	-	A	2.6	-	3.0	-
D	B	2.2	0.73	3.0	0.75	AB	2.1	0.81	2.0	0.67
TM	B	2.6	0.86	3.0	0.75	B	1.7	0.65	2.0	0.67
Cu	B	2.6	0.86	2.0	0.50	B	1.6	0.62	2.0	0.67
CCA	B	1.9	0.63	2.0	0.50	C	0.2	0.08	0.0	0.00
FA	AB	2.8	0.93	3.0	0.75	C	0.1	0.04	0.0	0.00
Ac	C	0.0	0.00	0.0	0.00	C	0.1	0.04	0.0	0.00

The major difference between above ground and soil contact testing is believed to be higher and more stable moisture content in the wood samples provided by soil contact exposure and direct contact to soil-inhabiting decay organisms, which frequently leads to an immediate onset of decay without any time lag at the beginning of exposure and to accelerated decay in general (e.g. Augusta 2007). For modified wood the most obvious difference between the two tests is how furfurylated wood only had initial decay in HDL, but when exposed in soil contact no significant difference was found between furfurylation and Scots pine sapwood. Results from long term field trials on modified wood are still sparse, but Larsson-Brelid *et al.* (2010) found that after 18 years in soil contact wood with acetyl content of about 20% was of the same magnitude as for CCA treated wood at a high retention level (10.3kg m^{-3}).

It is important to remember that the pick test rating normally used for field trials can suffer from some unintended evaluator bias, and the decay rating scale is not linear. Based on experience from Norwegian field trials samples can be rated 1 for a long time due to surface softening. Hence, one should be careful comparing materials with initial decay (i.e. rating 2 or below). In addition, if the degradation starts in the central part of a specimen the decay might not be detected with the pick test. This is often the case with copper treated wood (Humar and Thaler 2017).

Moisture tests

Table 4 provides the data from the 24 hour tests and capillary water uptake. For modified wood reduced water affinity is believed to be of great importance when it comes to mode of action mechanisms against wood deteriorating organisms while wood preservatives are mainly relying on a toxic effect (Hill 2006). This is also reflected in the results below, generally modified wood had lower moisture uptake than the preservative treated wood and untreated control.

$W24_{0\%RH}$: The highest values for desorption were determined for Scots pine sapwood, the lowest for acetylated wood. Acetylated wood had significantly lower desorption values than DMDHEU treated wood and furfurylated wood. No significant difference was found between DMDHEU treated wood and furfurylated wood or between thermally modified and acetylated wood.

$W24_{submersion}$: The highest value for liquid water uptake by submersion were found for Scots pine sapwood, the lowest for furfurylated wood. No significant difference was found between acetylated wood and DMDHEU treated wood or between thermally modified and DMDHEU treated wood.

$W24_{100\%RH}$: For water vapour uptake in water saturated atmosphere the highest value were found for copper HDO treated wood, the lowest for acetylated wood. No statistically significant differences were found between acetylated, furfurylated and thermally modified wood or between DMDHEU treated and thermally modified wood.

CWU: Capillary water uptake showed the highest value for Scots pine sapwood and lowest value for furfurylated wood. No significant difference was found between acetylated, thermally modified and DMDHEU treated wood or between furfurylated, thermally modified and DMDHEU treated wood.

Table 4

W24 tests and capillary water uptake

	$W24_{0\%RH}$		$W24_{submersion}$		$W24_{100\%RH}$		CWU	
	T-K	Mean [%]	T-K	Mean [%]	T-K	Mean [%]	T-K	Mean [g/cm^2]
SpS	A	17.2	A	93.2	ABC	17.2	A	0.34
Cu	ABC	13.7	B	79.2	A	24.1	B	0.18
CCA	ABC	14.6	CD	54.5	AB	23.3	C	0.13
D	AB	15.9	CD	52.2	BC	15.9	CD	0.11
FA	BC	12.3	E	23.6	D	7.6	D	0.09
TM	CD	10.2	C	60.6	CD	13.4	CD	0.11
Ac	D	6.5	D	45.0	D	6.5	C	0.12

In Table 5 the ranking of materials from all the different decay and moisture tests, based on ML_{MV}, decay rating or moisture performance, is provided. The highest performance within each test is given the value 1, the second best value 2, the third best value 3 and the lowest value 4. Then the sum from all tests was calculated for each material. The overall best performance was achieved for acetylated wood (score 16), while thermally modified wood (score 34), DMDHEU treated wood (score 34) and furfurylated wood (score 35) performed at a similar level in these tests. It need to be noted that this is not nessesarily a fair comparison of the treatments per se since all the treatment levels not nessesarily are fully compareable and because above ground treatment levels are exposed in soil contact. The aim of this paper is to compare test methods, not to rank materials.

The difference in material perfomance ranking between tests were also compared. Tests with similar ranking included: 1) $W24_{100\% RH}$ and HDL, and 2) mini block 11°C/70% RH and EN 252. The comparison also showed no difference between the three climates in the ENV 807 test or between 22°C and 16°C in the mini block test. The material ranking for CWU, $W24_{0\%RH}$ and $W24_{submersion}$ did not overlap with any of the other tests.

Table 5

Ranking of treatments from all tests, lowest performance (i.e. highest mean mass loss, decay rating or water uptake) to the left, highest performance (i.e. lowest mean mass loss, decay rating or water uptake) to the right.

	Ranking low → high			
Mini block 22°C/70%	TM	FA	D	Ac
Mini block 16°C/70%	TM	FA	D	Ac
Mini block 11°C/70%	FA	TM	D	Ac
ENV 807 25°C/80%	FA	D	TM	Ac
ENV 807 25°C/60%	FA	D	TM	Ac
ENV 807 10°C/85%	FA	D	TM	Ac
$W24_{0\%RH}$	D	FA	TM	Ac
$W24_{submersion}$	TM	D	Ac	FA
$W24_{100\%RH}$	D	TM	FA	Ac
CWU	Ac	D and TM		FA
HDL	D	TM	FA	Ac
EN 252	FA	TM	D	Ac

CONCLUSIONS

In the current study of UC3 retentions of modified Scots pine sapwood in different laboratory moisture and decay tests and two field trials acetylated wood provided the best overall performance. The durability performance of thermally modified wood, furfurylated wood and DMDHE treated wood varied between the different tests, confirming that the durability classification of a material, and the ranking between materials, is not a fixed value that can be based on one single test.

When comparing material ranking between tests W24 water vapour uptake in water saturated atmosphere and HDL + mini block 11°C/70% RH and EN 252 gave similar rankings.

The W24 test (i.e. moisture response) showed that the modified wood, as expected, had lower moisture uptake than the preservative treated wood and the control. However, the moisture behaviour could not fully alone explain the decay resistance of the materials.

Lowering the temperature did, as expected, slow down the decay rate, but it did not make much difference in the durability ranking of the materials.

In order to predict service life the authors recommend to combine decay and moisture data, e.g. as in Meyer-Veltrup *et al.* (2017). This is especially important for modified and preservative treated wood since different modes of action need to be considered.

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