IRG 24-60447

THE INTERNATIONAL RESEARCH GROUP ON WOOD PROTECTION

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NB

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Section 2. Wood Protection	WP 2.1. Wood preservatives	☐
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Section 3. Wood Modification	WP 3.1. Thermal modification	☐
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	WP 4.2. Service life	☐
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	WP 4.4. Analysis and Standards	x
Section 5. Sustainability and Environment	WP 5.1. Health and Safety Aspects	☐
	WP 5.2. Reuse and recyclability of treated or modified wood	☐
	WP 5.3. Environmental Impact	☐
	WP 5.4. Circular economy of wood products	☐

This paper is intended for the special session on:

☐ Mass Timber
☐ Circular Economy
☐ TBD

This paper will be presented as a(n):

x Oral presentation (15 minutes + questions)
☐ Poster presentation (including a 3-minute oral presentation)

This paper will most probably be presented by: Peter Klaas

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THE INTERNATIONAL RESEARCH GROUP ON WOOD PROTECTION

Section Y VVVVVVVVVVV

EMC as a quality control parameter for thermally modified wood

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ABSTRACT

While standards for quality performance of treated wood have been in place for many years, none have been developed for modified wood such as thermally modified wood (TMW). Only in one case has TMW been listed in 2018, by the Nordic Wood Preservation Council (NWPC).

A key component of a quality system is its control parameters, i.e. the metrics that are to be measured and controlled. Existing metrics for treated wood are not meaningful for TMW. In this paper we review literature and present an analysis of the lab and field test performance data used by the NWPC to list TMW. We propose Equilibrium Moisture Content (EMC) as a valid a reliable metric for TMW quality performance at a threshold value of EMC=6.

Thus, decay resistance of thermally modified wood is widely believed to be related to reduction of wood cell wall moisture capacity. We further explore the presence of a universal moisture threshold value, below which decay does not occur. The aim is to provide a scientific basis for quality performance measurement in industrial quality management systems and development of national standards.

Laboratory data are based on 900 specimens of 5 species/qualities, thermally modified at low, medium and high modification intensities. EMCs are measured and durability performance according to EN 350 is determined by means of two different procedures, CEN 15083-1 (fungal test) combined with EN 84 (accelerated ageing – leaching) and EN 73 (accelerated ageing – evaporation), respectively. We also report data from 6 years of accelerated testing in Borneo, after CEN 12037 (horizontal lap-joint).

Laboratory data reconfirm strong statistical correlations between moisture and durability performance as described in existing theory. Data also support the theoretical notion that a threshold EMC value exist, at which decay cannot progress. Surprisingly, the threshold, at same level of intensity, appear to be different for softwood and hardwood. Based on these insights, a threshold EMC value suitable for industrial quality control and national standards development is identified. 6 years of field data support and validate the statistically derived and proposed metric.

Keywords: Thermally Modified Wood, EMC, MEE, Quality Control, Nordic Wood Preservation Council, Field Data, Standard development

1. INTRODUCTION

As Sandberg et. al. notes (Sandberg et al. 2021: 218), it is important not only for quality control (QC) but also for scientific comparison of thermally modified specimens to be able to quantify the degree of modification achieved. However, for this quantification to be of any use in quality control (QC) and industrial quality management systems (QMS), it must also be a valid and reliable predictor of decay resistance and durability performance as well as practical in use.

Thybring (2013), see also Sandberg et al. (2021:62), in an excellent overview, compiles and analyses the performance of various types of modified wood reported in literature. Based on Moisture Exclusion Efficiency (MEE) and Anti Swelling Efficiency (ASE), he identifies for different modification technologies threshold values at which below decay cannot occur. In the case of thermally modified wood (TMW), as ASE is not meaningful, he suggests a MEE=42% as threshold value. Importantly, he discusses why this value, for TMW, may only apply for applications below water saturation point, i.e., for EN 350 Use Class 3 above ground. In TMW the fiber saturation point (FSP) may increase instead of the opposite because porosity is increased, even if the amount of accessible OH groups is reduced by thermal modification.

Experience from industry indicates that mechanical mass loss and porosity may depend on the specific modification technology, suggesting that mass loss and porosity is higher with pyrolysis than with hydrolysis type processes; further research may reveal if there is a porosity threshold value for TMT, at which below decay cannot occur in (near) water saturated conditions.

Thybring also discusses potential problems with using MEE as a measure for modification intensity and decay performance. From a practical, industrial view, EMC, on which MEE is based, seems like a more obvious candidate for quality control (QC) purposes. It is not dependent on an uncertain maximum moisture capacity value but is a manifest and simple measure. In industrial settings it can be measured directly in a practical way. It is also important that the measure is widely known and accepted in the industry, creating confidence in the QC processes.

Thybring's suggested TMW threshold of MEE=42% corresponds to an EMC of 7 at standard conditions 20^0C, 65% RH for although this will be species dependent.

A number of alternative measures have been suggested in literature, including color, mass loss, MEE, Electron spin resonance, nuclear magnetic resonance and near infrared spectroscopy. Some of these lack strong correlation between modification intensity and decay performance, while other seem more appropriate for laboratory use and are poorly understood in industry. See Willems et al. (2015) for a comprehensive review.

2. DATA AND EXPERIMENTAL METHODS

Laboratory data are from accredited tests performed by the Danish Technological Institute (DTI) in 2017. Tested species are Scots Pine heart wood, Scots Pine sap wood, Radiata Pine, Norwegian Spruce and European Beech.

Test sample size is N = 30 specimens with a total of 18 samples per species (3 intensities, 3 fungi and two durability testing procedures); the total number of samples in the study is 90 (only 30 are reported and used in the analysis because only the highest mass loss from each of the 3 fungi is used to determine durability performance according to the standard) and total number of specimens 2.700 (900 reported).

Modification was carried out at DTI in a lab autoclave enabling the WTT ThermoTreat 2.0^{pat} high pressure thermal modification process (see also Sosins et al. (2024)). Low, medium and high intensity modification was performed at 170, 180 and 190 ^{0}C, respectively, at pressures 4 bar above boiling point (8,0, 10,0 and 12,5 bar, respectively).

After modification EMC was quantified according to:

$$EMC\ (\%) = [(M_2 - M_1)/M_1]x100 \qquad (1)$$

Where M_1 is the oven-dry weight of the specimen after thermal modification and M_2 is the weight of the specimen before thermal modification, both at 20^{0}C temperature and 65% relative humidity. EMC was determined on the absorption curve, i.e. specimens were first dried down to zero moisture and then moisturized to equilibrium. This procedure influences moisture behavior so different (representative) samples were used for EMC determination and durability testing.

For each species, durability testing was performed according to CEN 15083-1: *Durability of wood and wood-based products – Determination of the natural durability again wood-destroying fungi, test methods – Part 1: Basidiomycetes* (16 weeks exposure to fungi), combined with two different ageing tests EN 73 – *evaporative ageing procedure* and EN 84 – *leaching ageing procedure*. 3 fungi were used, *Coniophora puteana*, *Poria placenta* and *Trametes versicolor*.

Durability performance was determined as the highest of the median mass losses (%) (ML) determined for test specimens exposed to each of the used fungi and evaluated after EN 350 (2016).

Table 1: EMC and Weight loss (WL) data from 5 species modified at 3 levels of intensity and using two different ageing procedures

1583-1+EN 84	**Species**	**Intensity**	**EMC (%)**	**WL(%)**
	Scots Pine Sap	Low	7,1	18
		Medium	6,1	2
		High	5	5
	Beech	Low	7	5
		Medium	6,6	3
		High	5,6	3
	Scots Pine Heart	Low	6,1	17
		Medium	5,6	1
		High	5,4	1
	Norway Spruce	Low	7	20
		Medium	5,9	3
		High	4,9	4
	Radiata Pine	Low	7	21
		Medium	6	3
		High	5,2	1
1583-1+EN 73	Scots Pine Sap	Low	7,1	18
		Medium	6,1	12
		High	5	2
	Beech	Low	7	2
		Medium	6,6	2

		High	5,6	2
	Scots Pine Heart	Low	6,1	17
		Medium	5,6	1
		High	5,4	1
	Norway Spruce	Low	7	20
		Medium	5,9	3
		High	4,9	1
	Radiata Pine	Low	7	18
		Medium	6	4
		High	5,2	1

Summary statistics	**Softwood and Hardwood**		
	Overall mean	6,0	7,0
	Mean EN 84	6,1	7,8
	Mean EN 73	6,0	6,9
	Hardwood only		
	Overall mean	6,4	2,8
	Softwood only		
	Overall mean	6,2	8,2

Field test data are from accredited test performed by DTI at their test site at University of Malaysia, Kuching, Borneo. The site has an annual average humidity of 80-90% at temperatures between 22 and 32^{0}C.

The samples were modified in January 2018, using the same process as for the laboratory tests, reported above. Three species, Beech, Scots Pine sapwood and Norwegian Spruce were modified at high intensity, in all cases resulting in EMCs below 6.

Field durability testing (ongoing) was initiated in May 2018, according to CEN 12037 (2003): *Wood preservatives – Field test method for determining the relative protective effectiveness of a wood preservative exposed out of ground contact – Horizontal lap-joint method*.

Durability performance was graded after the following system om table 2 below, and the durability performance of the samples is reported further below in Table 3.

Table 2: Field test performance rating system

Rating	Description	Definition
0	Sound	No evidence of decay
1	Slight attack	Visible signs of decay, but no significant softening or weakening of the wood
2	Moderate attack	Areas of decay (softened, weakened wood); typically not more than 3 cm^2 and to a depth of 2 to 3 mm
2+	Moderate attack +	Approaching 3, severe attack

3	Severe attack	Marked softening and weakening of the wood typical of fungal decay; distinctly more than 3 cm^2 affected and to a depth of 3 to 5 mm or 5 to 10 mm over a few cm^2
3+	Severe attack +	Approaching 4, failure
4	Failure	Very severe and extensive rot, joint member(s) often capable of being easily broken

Table 3: Median of Decay Rating in lap of thermally modified Beech, Scots Pine sap and Spruce

		Median of Decay Grading, Year					
	EMC	2019	2020	2021	2022	2023	2024
Beech	5.4	0	No	0	0	0	0
Scots Pine sap	5.8	0	ra-	0	1	1	1
Norwegian Spruce	5.8	0	ting	0	1	1	1
Untreated Beech	-	2	due to	3	3	4	4
Untreated Scots Pine sap	-	2	Covid	3	4	4	4
CCA	-	0		0	0	0	0

3. ANALYSIS AND DISCUSSON

From the summary statistics in tab. 1 above, it appears that, at similar EMC values, WL in Softwoods are higher than in Hardwoods. A simple t-test comparing EMC values of “hardwood only” and “softwood only” show no significant difference between them at t=1,36 and p=0,09 (one tailed), while the same test for the corresponding WL values confirms a significant difference at t=3,09 and p=0,002 (one tailed).

This is a surprising result and questions the existence of a universal modification intensity at which below fungal attack cannot occur. The decay resistance of modified wood is related to the reduction in maximum moisture capacity of the cell (Thybring 2013); but if different levels of decay resistance can be related to the same maximum fiber saturation point, then additional causes beyond moisture reduction influence decay resistance.

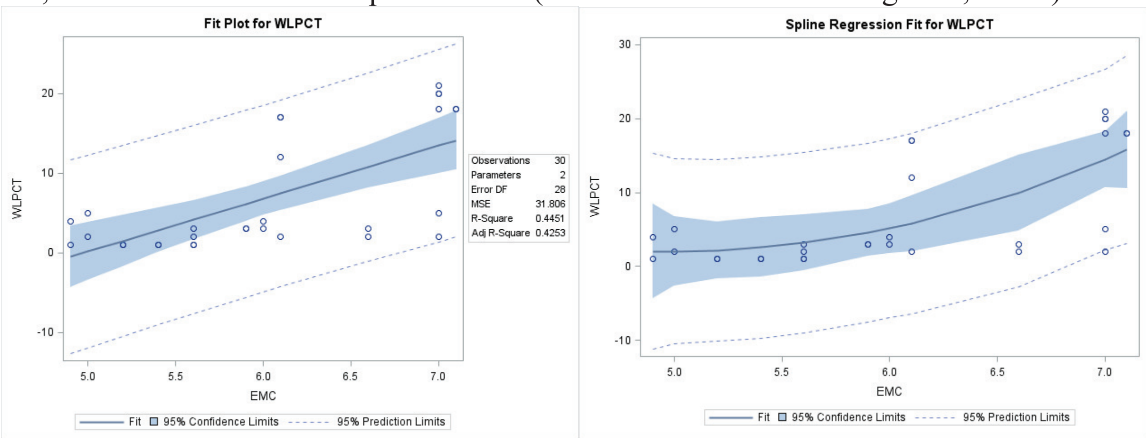
Based on the initial observation from the summary statistics, we proceed to examine which statistical model provides most information from the sample data.

For the results presented in Fig 1 and 2 below, the linear model is estimated using simple linear regression with WL as dependent variable and EMC as explanatory variable. Based on the same two variables, the nonlinear model is estimated using a spline regression. The two models are estimated using the SAS version 9.4 standard routines PROC REG and PROC TRANSREG respectively. Figures reported from both models are the F test for model significance, together with its p value, and the R-Square. While the F test and its p value indicate statistical significance, the R-Square indicates practical significance in term of model fit. The higher the R value, the better the model explains (fits) variation in data. These two regressions were performed for all observations (Fig. 1a,b hardwood and softwood) and for softwood only (Fig. 2a,b).

First we analyzed the total data sample N=30 including both softwoods and hardwood. This approach implicitly accepts the conceptual notion suggested in literature that a *universal*, in the meaning that the same threshold applies to all situations EMC threshold value exists across species. This has been assumed in research so far. In this scenario, the lower WL values of hardwood for similar EMC values seen in the summary data as discussed above, are interpreted as noise not providing information.

We apply two different models, a linear and a non-linear. The first linear model implicitly rejects the notion of a threshold EMC value, while the second non-linear model accepts the notion of a threshold EMC value.

Fig 1 a,b: Linear and nonlinear spline curves (Softwood and Harwood together, N=30)



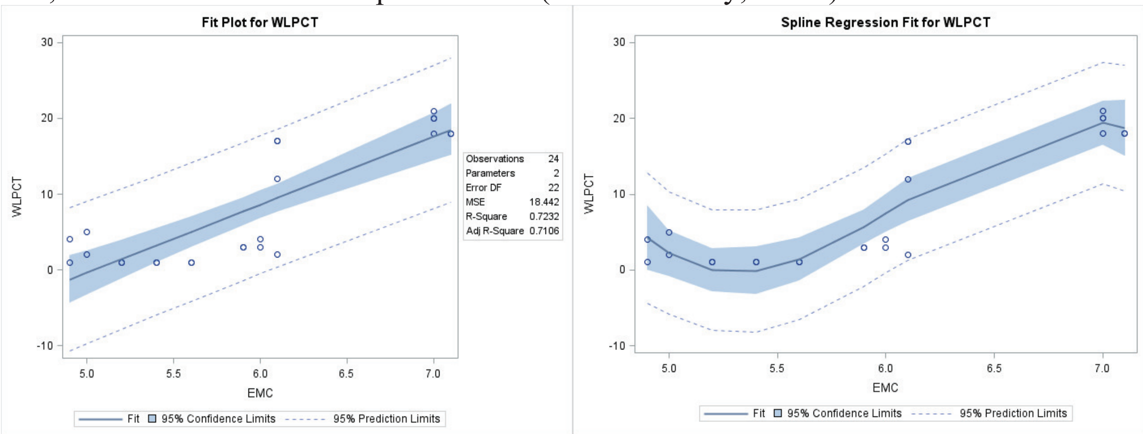
F=22.46; P<.0001; R^2=44.5% F=8.08; P=0.0006; R^2=48.2%

The results in Fig. 1a do not reject the notion that there is a linear correlation between EMC and WL, as the variation in the former explains 44.5% of the variation in the latter. Based on the difference in R^2 values there is a slight support for the nonlinear spline in fig.1b, indicating a nonlinear correlation of 48,2% in data; however, the difference in R^2 is marginal and does not offer clear support for the existence of a universal EMC value at which fungal attack can be prevented, represented by a shift in WL from high to low. This is surprising because a significant body of research claims that such a threshold should exist.

As suggested by the summary statistics, the reason for this may be that a universal EMC threshold does not exist, but instead that softwoods and hardwood have different thresholds and, as a result, are best described by two different statistical models. In this case, samples containing both softwood and hardwood data are not statistically representative because they belong to different populations and, as a result, cannot be concluded from.

Based on this, in the model below we separated softwoods and hardwoods and tested the model on the softwood data alone, N=24:

Fig. 2 a,b: Linear and nonlinear spline curves (Softwood only, N=24)



F=57.48; P<.0001; R^2=72.3% F=31.59; P<.0001; R^2=82.6%

Despite reduced sample size and corresponding reduction in degrees of freedom, the models shown in Fig. 2 a,b provides high correlation between EMC and WL without loss of statistical significance (P<0,0001). This lends support to the hypothesis that the expected theoretical correlation between EMC and WL is not universal, but specific to hardwood and softwood (it may even be species specific).

Comparing R^2 values between Fig 2.a (linear correlation, no threshold value) and Fig 2.b (non-linear correlation with threshold value, variation in data is best explained and predicted by the statistical model in Fig. 2.b. This model represent (Thybring's 2013) hypothesis that a threshold value of modification intensity, represented here by EMC, does exist. However, it does not seem to be universal but specific to wood species.

Hardwoods in general have higher levels of hemicelluloses compared to softwood. The largest residual component from the hydrolysis of hemicelluloses are organic acids, primarily acetic acid. It can reasonably be hypothesized that, when using a high pressure thermal modification process where the residual acids are kept in fluid form within the wood, the amount of residual acids after modification are in general higher with hardwoods than in softwoods. Since acids, such as tannins, are well known contributors to natural wood decay resistance, it also follows that the increased residual acid levels in TMW hardwood may cause additional decay resistance, beyond reduction in accessible hydroxyl groups.

The final test focuses on the development of an EMC based quality control parameter value which can be used for industrial Quality Control (QC).

As discussed above and in literature, ASE, MEE or EMC threshold values at which decay cannot occur may be subject to significant variation. Such variation and heterogeneity create challenges in developing quality and industry standards, as well as uncertainty in market and consumer product quality confidence. Is it possible to identify an industrially useful QC criteria based on a universal EMC value, at which the prevention of decay can be assured?

Thybring et al. (2013:92) suggested a 42% MEE threshold for TMT. If we assume that for Scots Pine, at 20^0C and 65%RH, EMC is 12%; then, at 42% MEE, after modification EMC=6,96 so that a 42% MEE corresponds to EMC=7 at ambient conditions.

Tab. 2 below displays a set of WL values which illustrate the rate of expected defects at a given level of quality, based on common Statistical Process Control (SPC) principles.

Table 2: Distribution of Weight Loss (WL) (%) for EMC <= 7%.

	Hardwood and Softwood	Softwood only
N	28	22
Mean	6.250	7.182
Standard deviation	7.064	7.719
P(WL>5)(%)	57.02	61.13
P(WL>6)(%)	51.41	56.08
P(WL>7)(%)	45.77	50.94
P(WL>8)(%)	40.22	45.78
P(WL>9)(%)	34.85	40.69
P(WL>10)(%)	29.78	35.75

Assuming normal distributions for WL with the means and standard deviations as indicated in tab. 2 for the two groups (hardwood and softwood together and hardwood only), the probabilities of WL exceeding selected thresholds (e.g. 5%, 6%, 7% etc.) are as reported in Table 2 above. Thus, the probability for WL exceeding a threshold value of 5% WL, is 61,13%. In practical terms, more than 62 boards out of 100 would display a weight loss of more that 5% from fungal attack.

Translating the results in Tab. 2 above, for a modified product quality level of maximum 5% WL, corresponding to Use Class 1 after EN350, the expected rate of defects is 61,13 at EMC=7. For example, in 100 boards (siding, decking) 61,13 boards can be expected to fail on average. At EN350 Use Class 2 quality performance level, the expected rate of failure is 35,75 %. This may be overestimated because of the relatively small sample size. Larger sample sizes may be expected to reduce standard deviation and cause expected failure rate to fall. However, our data suggest that threshold value EMC=7 is too high for QMS purposes, especially in the absence of supporting field test data.

Looking at data in table 1, WL values over 5 are not observable for EMC< 6%, corresponding to MEE > 50%.

Table 3: Distribution of Weight Loss (WL) for EMC <6

	Hard and Softwood	Softwood only
N	14	12
Mean	2.071	2.000
Standard deviation	1.328	1.414
P(WL>=5) (%)	1.37	1.69
P(WL>=6) (%)	0.15	0.23
P(WL>=7) (%)	0.01	0.02
P(WL>=8) (%)	0.0004	0.001
P(WL>=9) (%)	<0.0001	<0.0001
P(WL>=10) (%)	<0.0001	<0.0001

Assuming normal distributions for WL with the means and standard deviations as indicated for the two groups (hardwood and softwood together and hardwood only), the probabilities of WL exceeding selected thresholds are as reported in Table 3 above. Thus, the probability for WL exceeding a threshold value of 5% WL is 1.69%.

Translating the results in tab. 3 above to Statistical Process Control (SPC) terms, for a modified product quality level of maximum 5% WL, the expected rate of defects is 0,0137. Or, for example, in 100 boards (siding, decking) 1,37 boards can be expected to fail. The rate may be overestimated caused by relatively small (but statistically significant) sample size. With larger sample size standard deviation may reduce causing a decrease in the expected defect rate.

In conclusion, analysis of quality performance in terms of WL from fungal attack in a sample including 5 wood species/qualities, modified at three different intensities and performance tested using two different ageing procedures suggest that, for TMT, a universal quality parameter value EMC=<6 should ensure good durability performance quality, while EMC=<7 may well be too high. Translated to MEE, previous research has suggested 42%; this may be too low and should be adjusted to MEE=<50%.

The conclusion is further validated from field test data reported above; in the test, Beech, Scots Pine sapwood and Norwegian Spruce, all modified to EMC values below 6, display good durability performance in a climate where the reference samples failed after 3 years exposure. Secondary field test data are required for listing in industrial bodies (e.g. AWPA, WPC and NWPC) and standards (e.g. EN, BS, DIN).

As an example, the Nordic Wood Preservation Council (NWPC) is using the EMC=<6 criterion for their lab and field test requirements for their Thermally Modified wood QMS standard ((NWPC List no. 103 (2024)).

4. CONCLUSION

Based on extant literature this paper proposes EMC as quality metric for TMW and argues that this metric is more reliable than MEE, because EMC is a more direct proxy for fiber moisture capacity than MEE (which is a relative metric).

Statistical analysis was performed on comprehensive laboratory and field data, previously used by the Nordic Wood Protection Association for listing TMW. The results reconfirmed and validated extant scientific research on the relation between cell wall moisture capacity, as measured by EMC, and durability performance.

The analysis provided additional insights, as data did not justify the presence of a universal, one to one relationship between moisture capacity, measured as EMC, and decay resistance. Instead, for comparable levels of EMC, hardwoods have significantly lower weight loss from fungal attack than softwoods.

It is hypothesized that increased levels of residual acids in hardwoods cause additional decay resistance compared to hardwood, caused by their higher natural levels of hemicelluloses. Extant literature suggests a modification threshold level below which decay cannot occur at MEE=42%; this threshold value was not confirmed by our data. An alternative threshold level at MEE=>50%, corresponding to EMC=<6%, was accepted.

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