EMC as a quality control parameter for thermally modified wood

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Abstract

Decay resistance of thermally modified wood is widely believed to be related to reduction of moisture capacity in the wood cell. This paper further explores the presence of a universal moisture threshold, below which decay does not occur, providing a scientific basis for industrial quality management systems and development of national standards. 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) with EN 84 (accelerated ageing – leaching) and EN 73 (accelerated ageing – evaporation), respectively. 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, is different for softwood and hardwood as well as lower than previously reported. Based on these insights, a threshold EMC value suitable for industrial quality control and national standards development is identified.

Introduction

As (Sandberg et al. 2021: 218), it is important not only for quality control (QC) but also for scientific comparison of THM 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.

(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 TMT, as ASE is not meaningful, he suggests a MEE=42% as threshold value. Importantly, he discusses why this value, for TMT, may only apply for applications below water saturation point, i.e., for EN 350 Use Class 3 above ground. In TMT the fiber saturation point (FSP) may actually 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 be 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 TMT threshold of MEE=42% corresponds to an EMC of 7 at standard conditions 20^0C, 65% RH.

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.

Data

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 ThermoTreat 2.0[pat] high pressure thermal modification process. 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

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

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.

For each species, durability testing was performed according to CEN 15083-1 (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.

Table 1: EMC and WL data from 5 species treated at 3 intensities and using two ageing procedures

	Species	Intensity	EMC	WL%
1583-1+EN 84	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

Analysis and discussion

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.

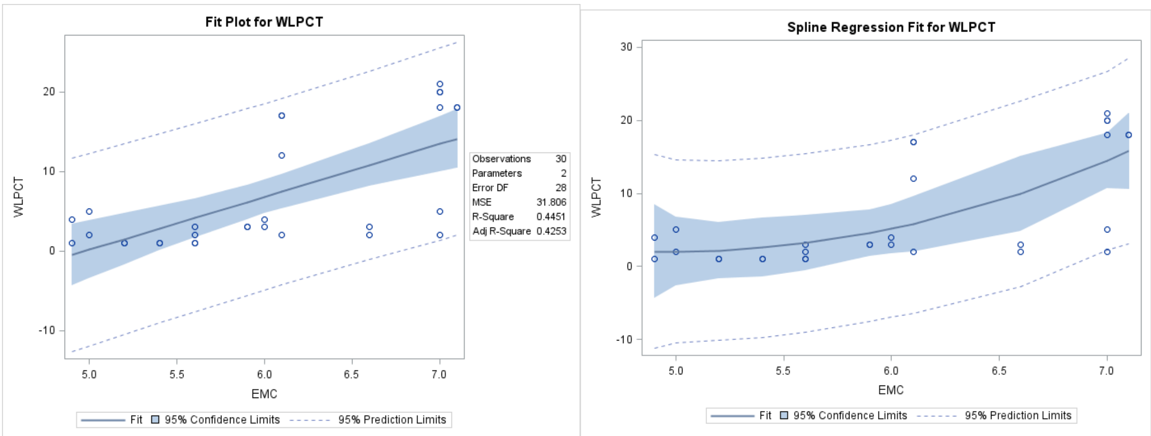
Based on the initial observation from the summary statistics, we proceed to examine which statistical model provides most information from the sample data.

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. These two regressions were performed for all observations (Fig. 1a,b hardwood and softwood) and for softwood only (Fig. 2a,b).

First we examine the total data sample N=30 including both softwoods and hardwood. This approach implicitly accepts the conceptual notion suggested in literature that a *universal* EMC threshold value exists across species. In this case, 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 non-linear spline curves (softwood and hardwood together, N=30)



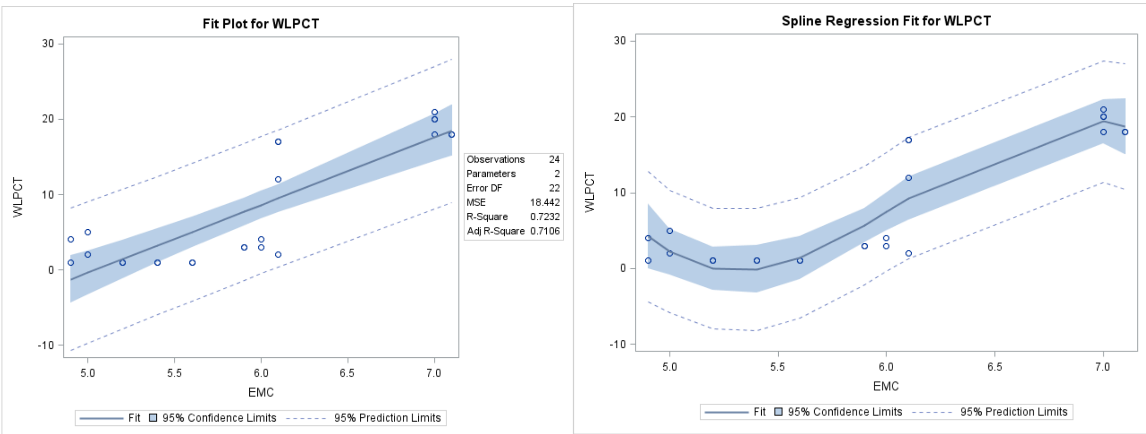
F=22.46; P<.0001; R^2=44.5%   F=8.08; P=0.0006; R^2=48.2%

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. 2a,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 TMT 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 2013:92) suggested a 42% MEE threshold for TMT. At 20^0C and 65%RH, EMC is 12%; 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) metrics.

Table 2: Distribution of WL for EMC <= 7.

	H and S	**S 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%.

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.

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

Table 3: Distribution of WL for EMC < 6.

	H and S	S 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 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%.

As an example, the Nordic Wood Preservation Council (NWPC) is using the EMC=<6 criterion for their Thermally Modified wood QMS standard.

Conclusion

Decay resistance in thermally modified wood is widely believed to be related to reduction of moisture capacity in the wood cell. Based on analysis of data from 900 specimens and 5 species/qualities, this relation is reconfirmed. However, analysis revealed that there is not 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. Extant literature suggests a modification threshold level below which decay cannot occur at MEE=42%; this threshold value was rejected, while an alternative threshold level at MEE=>50%, corresponding to EMC=<6%, was accepted. EMC is a familiar and accepted measure in industry and convenient for practical use, and the use of EMC=<6 as a general quality parameter for developing national standards and industrial quality management systems, is discussed.

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