Controlling moisture content of wood samples using a modified soil-pan decay method

Jerrold E. Winandy*
Simon F. Curling
Patricia K. Lebow

Abstract

In wood, the threshold level below which decay cannot occur varies with species or type of wood product and other factors such as temperature, humidity, and propensity of exposure or service-use to allow rain-induced wetting and subsequent drying. The ability to control wood moisture content (MC) during laboratory decay testing could allow research on the moisture thresholds required for the initiation of wood decay over a range of moisture conditions. In this study, we modified the soil-pan decay test method to control wood MC by controlling the vermiculite moisture levels. A model was developed to understand and control wood MC when using this modified soil-pan decay method.

A major factor in the initiation of decay of wood by fungi is the moisture content (MC) of the wood substrate (Lea 1992, Straube 1999, Rapp et al. 2000). Wood MC of 20 percent is generally regarded as the threshold value below which decay cannot occur (Sherwood and Stroh 1989, British Standards Institution 1992, Cassens et al. 1995), although some work has suggested that decay does not occur below 25 percent wood MC (Viitanen and Ritschscoff 1991, Rapp et al. 2000). As wood MC increases, the rate of decay should theoretically increase until moisture availability is no longer a limiting factor. Excessive wood MCs may reduce decay activity through water logging the wood, which prevents gaseous exchange (Rayner and Boddy 1988). Other factors that affect fungal growth, e.g., temperature, fungal and wood species, heartwood/sapwood ratio, and extractives, will also affect the rate of decay.

The exact MC required before decay will occur, termed the threshold MC, varies with the type of wood material used (Lea 1992). Obviously, maintaining the material below its threshold MC will prevent decay. However, under typical construction and service conditions, this recommendation cannot always be maintained and MCs occasionally rise above the threshold level. The length of time that wood MC is above the threshold level is therefore critical for long-term structural performance. Understanding decay progression and the rate of decay at MCs above the threshold, but below the optimum, may therefore allow determination of the relative risk of decay under varying conditions. This fundamental knowledge is critical for future attempts to model decay and durability.

That wood strength is significantly affected by decay has long been recognized and studied (Cartwright et al. 1931, Mulholland 1954, Kennedy 1958). The development of laboratory methodologies to access strength loss in wood undergoing incipient decay has recently shown that strength-related assessment of decay is a more sensitive measure than is the determination of weight loss, the commonly used measure for assessing decay (Winandy and Morrell 1993, Curling et al. 2002a, 2002b). A ground-contact-simulating test method has been developed that uses strength as the decay criterion. It may be possible to control the test conditions to produce a range of wood MCs and temperature conditions that could be used to determine the time to initiation and the rate of decay as a function of temperature and available

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The authors are, respectively, Project Leader-Performance Engineered Composites, Post-Doctoral Fellow, and Mathematical Statistician, USDA Forest Serv., Forest Products Lab., One Gifford Pinchot Dr., Madison, WI 53705. The authors would like to acknowledge the technical assistance of Richard Bergman and Rita Rentmeester of the Forest Products Lab. This paper was received for publication in February 2004. Article No. 9834.
*Forest Products Society Member.

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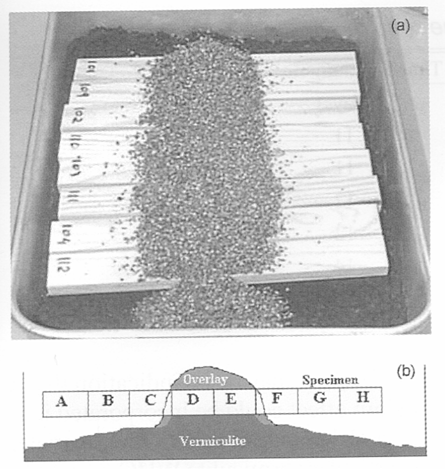

Figure 1. — Wood-media moisture test: a) experimental set-up; b) schematic showing location of eight sub-samples after removal and cutting.

moisture in the wood. The study reported here was an attempt to adapt the FPL soil-pan decay methodology to provide this range of conditions.

Method and materials

This study was conducted in three phases: 1) initial study of how wood MC changes relative to available moisture in soil-pan media; 2) model development; and 3) model verification.

Phase 1. Wood - media moisture relationships

Southern pine (*Pinus* spp.) sapwood test specimens (20.0 by 2.5 by 0.95 cm) clear of knots, damage, or defect were prepared and air-dried to approximately 12 percent MC.

The water-holding capacity (WHC) of the vermiculite media was determined from six random samples using the method described in ASTM D 2017-91 (ASTM 1999). One liter of vermiculite (horticultural grade) (Scotts, Marysville, Ohio) was placed in a lidded aluminum pan (330 by 230 by 82.5 mm) so that the bottom of the pan was covered. The vermiculite was then formed into a ridge, approximately 70 mm wide, running the length of the long axis. Deionized water was added to bring the MC of the vermiculite up to 15, 25, 50, 73, 75, 95, or 98 percent of the predetermined WHC of the vermiculite media. Multiple exposures, or batches, were also performed at 15, 50, and 73 percent WHC. Within each batch, six replicate pans were evaluated at each tested condition.

The specimens and pans were steam sterilized for 45 minutes at 105°C. The wood specimens were then placed, using aseptic technique, onto the pre-formed ridge in the vermiculite. Seven specimens were placed into each pan at each of the seven moisture conditions evaluated. The center sections of the specimens (middle 5 cm) were then covered to a depth of 2 cm with sterile vermiculite **(Fig. 1a)** at an MC equal to that of the base vermiculite, with the following exceptions: two tests used a 50 percent WHC overlay of media on a 75 percent WHC base (73% WHC equivalent), one test used a 75 percent WHC overlay on a 100 percent WHC base (98% WHC equivalent), and one test used a 50 percent WHC overlay on a 100 percent WHC base (95% WHC equivalent). The sterile, uninoculated pans were placed into a controlled conditioning room maintained at 25°C and 70 percent relative humidity for 3 weeks. As our objective was to study moisture sorption in wood, specimens were kept sterile over the course of the test.

A specimen was aseptically removed from each pan after 0, 1, 2, 4, 7, 14, and 21 days of exposure. Deviations in exposure times were necessary for some batches because of time constraints. These replicate specimens at each exposure condition were immediately cut into eight equal 2.5-cm-long sections (labelled A to H, **Fig. 1b).** The sections were quickly weighed, ovendried at 103°C, and reweighed to determine MC at the time of removal from vermiculite-media contact. Sections D and E (hereafter termed the center sections) represent the sections that initially absorbed water and should be most prone to decay. Sections A and H represent the ends of the specimen. Sections B and G, then sections C and F, were progressively taken from the ends toward the center.

Phase II. Development of model for moisture absorption

Wood MC data were used to develop a predictive model to estimate the wood (center section) MC in terms of percentage of vermiculite WHC. Simple asymptotic regression models for moisture uptake were evaluated for their ability to describe the collective "moisture absorptive" behavior of the exposed wood specimens over time. A step-wise approach was followed similar to that outlined in Pinheiro and Bates (2000) for nonlinear model fitting, using the nonlinear mixed-effects modeling package.

Phase III. Verification of model for moisture absorption

To determine the accuracy of the model developed in Phase II, the experiment was partially repeated. In this round of tests, a second set of southern pine (*Pinus* spp.) specimens was used to determine the accuracy of the predictive model. The Phase II model was inverted to predict the vermiculite WHC needed to produce 26, 29, and 32 percent wood MC.

For each of the three WHC conditions tested, 16 trays (8 replicates each for Sets A and B) were set up; each tray contained 7 specimens in two sets (A and B). At each sampling time (every 3 to 4 days), one specimen was removed from each of the eight replicate trays of Set A or Set B. For example, starting on day 3, Set A was sampled weekly to provide sampling times of 3, 10, 17, 24, 31, 38, and 56 days; starting at day 7, Set B was sampled weekly for sampling times of 7, 14, 21, 28, 35, 42, and 70 days. In all other respects, the experiment was conducted as in Phase II, except that wood MC was monitored over a longer period (12 weeks).

Results

Phase 1. Data analysis

Because the dry wood specimens absorbed moisture from the vermiculite bed and overlay, the central portion of the specimens (sections D and E) was progressively wetted; moisture then wicked out toward the ends. Examples of this progression in moisture profile within samples exposed at 75, 25, and 15 percent WHC are shown in **Figure 2.** The middle section (sections D and E), which was in direct contact with the vermiculite overlay, had the highest infusion of moisture and, eventually, had the highest final MC. The MC of sections D and E increased rapidly over time until a peak moisture was achieved. Moisture entering the wood in the area of sections D and E progressively wicked out towards the ends of the samples (sections A and H).

All moisture conditions tested exhibited a similar progression of wetting and wicking moisture out toward the specimen ends, varying primarily in regard to rate and magnitude of absorption. The MC of the intermediate sections that

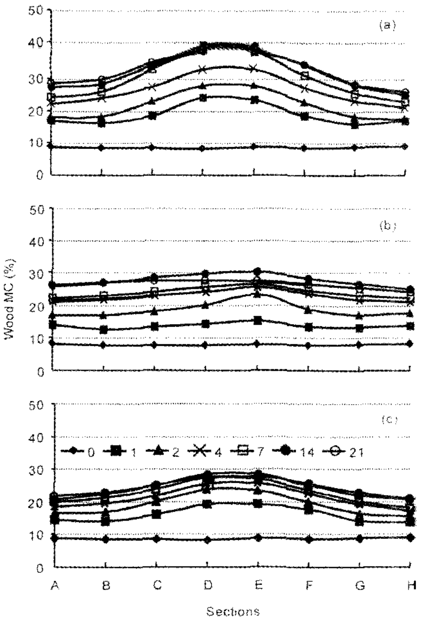

Figure 2. — MC profile of sample exposed at: a) 75 percent; b) 25 percent; and c) 15 percent WHC over time. Sections A and H are 2.5-cm sections near ends; sections D and E are 2.5-cm center sections (Fig. 1b) of one 20-cm-long specimen (Fig. 1a). Sections were exposed for 0 to 21 days, removed, and ovendried.

Table 1. Observed peak wood MC and time required to reach peak.

Vermiculite MC (% WHC)	Maximum wood MC (%)	Time to maximum MC (days)	Equilibrium MC
15	29	11	28
25	30	11	28
50	34	12	34
73	35	12	35
75	39	14	39
95	64	21	64

were not in contact with the vermiculite but were not at the ends also increased, but it did not increase as rapidly nor to the same level as did the MC of sections D and E. Also note that after the sections reached maximum MC (in about 14 days), the progressive wicking of moisture from the middle of the specimen (in contact with the vermiculite) sometimes lowered the eventual 21-day mid-section equilibrium MC to slightly below the 14-day maximum, as shown at 25 percent WHC **(Fig. 2b)** and 15 percent WHC **(Fig. 2c).**

The MC profiles of wood exposed to other vermiculite WHC levels each exhibited progressively similar trends to those shown in **Figure 2** for 75, 25, and 15 percent WHC. The observed peak wood MC and the time to obtain that peak varied **(Table 1).**

Phase II. Model development

The model was developed in three steps. First, a three-parameter nonlinear model was individually fit to each of the 10 WHC data sets tested in Phase I (Eq. **[1]).** Initial asymptotic regressions were individually fit to mean MC responses at the center of stakes over time and within each batch of vermiculite. For a given batch of vermiculite at WHC *w*, the MC model is expressed as:

$$MC_w = \beta_{w,1} + (\beta_{w,2} - \beta_{w,1}) \times \exp(-\beta_{w,3} \times time) + \varepsilon \qquad [1]$$

where:

MC_w = percentage of MC at center of stake

$time$ = days of exposure

$\beta_{w,1}$ = maximum MC (horizontal asymptote) as *time* increases

$\beta_{w,2}$ = MC at time zero

$\beta_{w,3}$ = rate of moisture uptake

$\varepsilon \sim N(0,\sigma^2)$ = normally distributed random error

In fitting Equation [1] to the data, the rate of moisture uptake is constrained for positivity by setting $\beta_{w,3} = \exp(\alpha_w)$, so that $\alpha_w = \ln(\beta_{w,3})$. After developing a series of parameter estimates, $\hat{\beta}_w = (\hat{\beta}_{w,1}, \hat{\beta}_{w,2}, \hat{\beta}_{w,3})$, using Equation **[1]** for each of the 10 WHCs evaluated, these estimates were reviewed for similarities and differences among the batches of vermiculite. Graphs indicated that the variability seen in the parameters could partially be explained by changes in WHC of the vermiculite, either as a covariate or as random effects.

The parameter estimates and fit statistics for each model are listed in Table 2. As indicated, the fits to nonlinear Equation **[1]** are satisfactory, with observable discrepancies primarily in the batches of vermiculite treated to higher WHC. The estimated maximum MC, given in Figure 3, shows individual parameter estimates encompassed in 95 percent confidence intervals. Within the sub-graph for each parameter estimate, overlapping confidence intervals can indicate a common underlying parameter, such as the MC at time zero ($\beta_{w,2}$). Non-overlapping confidence intervals, such as occurs for $\beta_{w,1}$, may be an indication that a random effect could be incorporated into the model or that an underlying covariate, in particular vermiculite WHC, may help explain the batch-to-batch variation.

The next step of model development was to fit a model with the three main parameters each associated with a particular random effect to accommodate various WHC in the media and batch-to-batch variation. Using this model then allows us to differentiate the effects of WHC on maximum wood MC (β_1) and rate of moisture uptake (β_3) from the random effects.

The ensuing nonlinear model developed had three primary parameters that modeled mean effects (initial and maximum wood MC, rate of uptake) and also estimated random effects associated with each primary parameter to capture batch-to-batch variability:

$$MC = \phi_1 + (\phi_2 - \phi_1) \times \exp(-\phi_3 \times time) + \varepsilon \qquad [2]$$

where:

$\phi_1 = \beta_1 + b_1$ = maximum MC (horizontal asymptote)

$\phi_2 = \beta_2 + b_2$ = common MC at time zero

$\phi_3 = \exp(\beta_3 + b_3)$ = rate of moisture uptake

$(b_1, b_2, b_3) \sim N((0,0,0), \mathrm{diag}((\sigma_1^2, \sigma_2^2, \sigma_3^2))$ = normally distributed random effect describing variability between batches of vermiculite at same WHC

$\varepsilon \sim N(0,\sigma^2)$ = normally distributed random error

Analysis of the pair-wise scatterplots (not shown) of the individual nonlinear estimates from Equation **[1]** did not indicate correlations between parameter estimates. This led to Equation **[2],** which provides a logical method with which to begin to group, and thereby in-

Table 2. — Parameter estimates for individual models of MC fit by Equation [1].[a]

WHC (%)[b]	Estimates for parameters				
w	$\beta_{w,1}$	$\beta_{w,2}$	$\beta_{w,3}$ (exp(α_w)	r^2	RSE
15(1)	27.6	8.7	0.77	0.993	0.73
15(2)	27.8	8.4	0.89	0.978	1.33
25	27.7	7.6	0.47	0.997	0.50
50(1)	33.1	11.1	0.36	0.996	0.64
50(2)	34.4	11.9	0.36	0.975	1.57
73(1)	34.6	11.2	0.32	0.996	0.64
73(2)	35.3	12.1	0.30	0.983	1.34
75	38.7	9.4	0.54	0.984	1.72
95	53.3	11.6	0.82	0.947	4.52
98	55.5	12.3	0.64	0.939	5.10

[a] $R^2 = 1 - \sum (MC - \hat{M}C)^2 / \sum (MC - \overline{MC})^2$. RSE = residual standard error.

[b] Batch numbers are in parentheses.

Table 3. — Parameter estimates for MC for all data fit by Equation ***[3].***

Parameter	Estimate	Standard error	p-value (based on t_{56})
$\phi_1 = \beta_{11} + \beta_{12} w^2 + b_1$			
β_{11}	25.60868	1.756806	< 0.0001
β_{12}	0.00273	0.000336	< 0.0001
ϕ_2	10.43043	0.700734	<0.0001
$\phi_3 = \exp(\beta_{31} + \beta_{32} w + \beta_{32} w^2)$			
β_{31}	0.26313	0.322740	0.4184
β_{32}	-0.04929	0.011574	0.0001
β_{33}	0.00046	0.000093	< 0.0001
σ_1	3.078006		
σ	2.175690		

terpret, the individual models. We evaluated plots of the resulting estimated random effects for each batch from fitting Equation **[2]** against WHC of the associated batch of vermiculite. This evaluation of the random effects associated with MC at time zero (62) relative to its estimated value indicated that it was not necessary to include a random effect for this parameter. However, random effects of a larger magnitude appear to be associated with WHC of vermiculite. The random effects associated with the asymptote parameter appeared to have an increasing pattern; random effects associated with the uptake rate indicated a possible decline, followed by an increase or flatness through midpoints, with higher uptakes at the extremes.

This analysis indicates that the inclusion of each batch of vermiculite WHC as a covariate might explain much of the pattern in random effects. Several models were evaluated for different forms of covariate inclusion, with the final model including quadratic functions for explaining random effects. This eliminated the need for the random effect b_3 for the uptake parameter and reduced the estimate of variance associated with the random effect b_1 for the asymptote parameter.

This analysis led to the development of Equation **[3],** which represents a versatile model to predict wood MC as a function of the percentage of WHC of the media and duration of exposure to that soil-pan media. Equation **[3]** adjusts parameter estimates for the asymptote and rate of moisture uptake for a given WHC:

$$MC = \phi_1 + (\phi_{j\,2} - \phi_1) \exp(-\phi_3 \times time) + \varepsilon \qquad \textbf{[3]}$$

where:

$w =$ WHC

$\phi_1 = \beta_{11} + \beta_{12} w^2 + b_1$
= maximum MC (horizontal asymptote) at w as *time* increases

$\phi_2 =$ common MC at time zero

$\phi_3 = \exp(\beta_{31} + \beta_{32} w + \beta_{33} w^2)$
= rate of moisture uptake at w

$b_1 \sim N(0, \sigma^2_1)$
= normally distributed random effect describing variability between vermiculite batches at same w

$\varepsilon \sim N(0, \sigma^2)$
= normally distributed random error

Parameter estimates for Model 3 are given in **Table 3.**

Since the performance of the individual models fit to Equation **[1]** is very good, these models provide a benchmark with an overall residual error of 2.36. Model 2 is the first-stage random effect model, which is further refined in the second-stage random effect model (Model 3). Model 2 has a residual error estimate of 2.23 and error estimates of 9.51 associated with the asymptote random effect, 0.28 associated with the uptake rate random effect, and 5.5×10^{-6} associated with the initial MC random effect. Model 3 has a residual error estimate of 2.18 and asymptote random effect error estimate of 3.08.

Residual plots indicated no systematic trends and were judged as satisfactory in our opinion. While outliers were observed at the tails for extreme MC in the vermiculite media as WHC was increased this probably indicated that as the vermiculite media was progressively manipulated to higher and higher WHC levels, fully saturated sub-zones of vermiculite were established. Wood specimens in direct contact with those subzones would gain more moisture than expected. The performance of the model is illustrated in **Figure 4.**

The fixed effect portion of the model (obtained by assuming random effects are zero) provides the ability to predict future average MC in the center of stakes exposed to vermiculite held at other

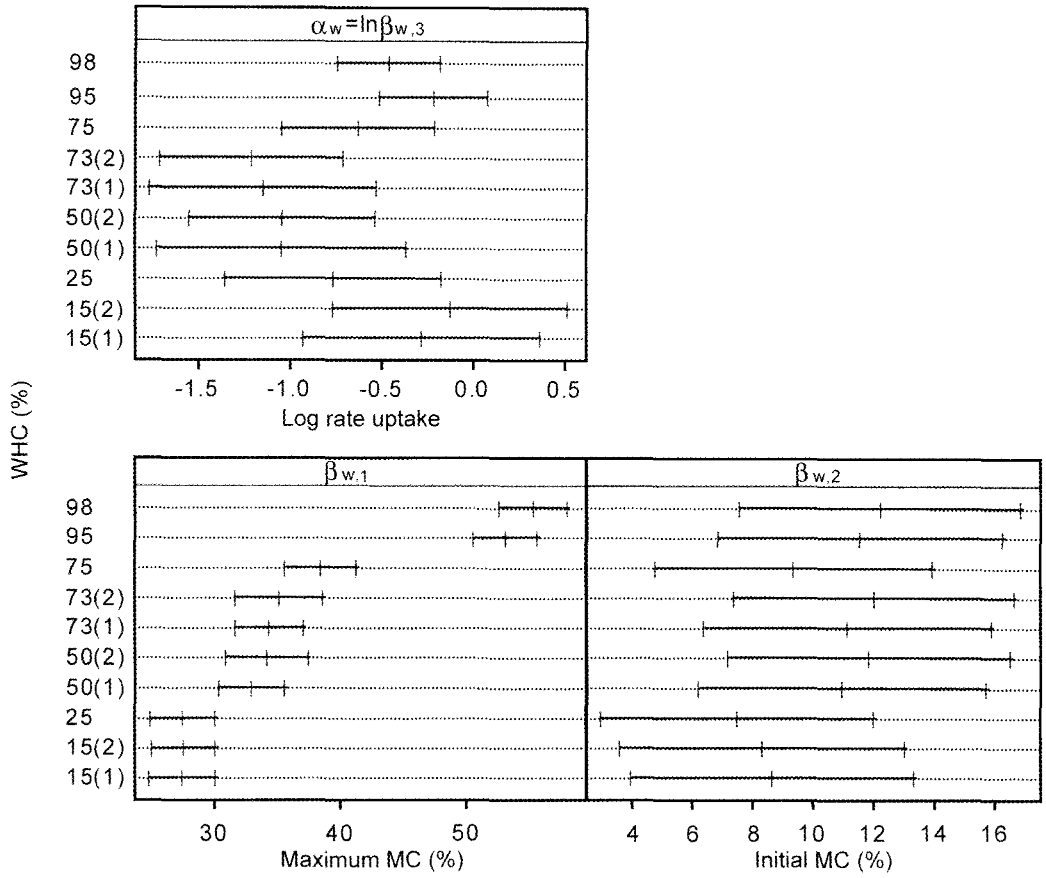

Figure 3. — Parameter estimates and 95 percent confidence intervals for individual nonlinear regressions (from Eq. ***[1]****) for each batch of vermiculite where* $\beta_{w,1}$ *= maximum MC (wood),* $\beta_{w,2}$ *= initial MC (wood) and* $\beta_{w,3}$ *= rate of water uptake in wood.*

WHCs. **Figure 4** illustrates the fit of the fixed effect portion of the model (estimated population mean obtained by setting random effects to zero) as well as the fixed plus random effect model (obtained by setting random effects to calculated value).

Phase III. Model verification

Performance was evaluated for an independent set of data based on visual fit **(Fig. 5).** Although the MC values in this independent set of data were calculated from entire stakes as opposed to the center of the stakes, model predictions based on the assumption that random effects are zero are well within expectations, especially when extrapolating beyond the exposure range of the developed models. Extrapolating beyond these MCs and species has not been fully studied and should not be done without caution. Our work continues to better define the models for pine, and we hope to eventually relate these models to other species.

Discussion

The data showed that by adjusting the initial MC of the vermiculite, the peak MC of the wood samples could be adjusted and maintained for a period. The data also show that the vermiculite overlay did indeed raise the MC of the center sections (D and E) of the specimen in comparison to the outer sections **(Fig. 1).** This also explains why the localized (sections D and E) MC was higher than that of the gross specimen. This is important in that during decay tests the fungal action will be localized in this area, making subsequent strength testing more accurate (Curling et al. 2002a, 2002b).

One problem experienced in these tests was dehydration of the system, especially at lower initial moisture conditions. At the lowest moisture level (i.e., WHC 10.5% for pine MC of 26%), wood MC reached its peak at about 2 weeks, but then rapidly dropped after about 3 weeks. However, the peak MC was maintained for about 8 to 10 weeks at the two higher moisture levels (i.e., WHC 21.5% and 42% for pine MC of 29% and 32%, respectively). This loss in overall moisture within the decay chamber may have been due to the repeated opening of the pans during periodic specimen harvesting, which allowed moisture to leave the system. This moisture loss would obviously have the greatest effect on the system at the lowest moisture condition (as shown by the data). In subsequent investigations, it was possible to determine the rate of moisture loss by monitoring mass loss and subsequently to replenish the water lost during each pan-opening sequence by adding 7 to 12 mL of sterile water (depending on desired wood MC and media WHC) to the vermiculite media whenever the pans were opened.

Although obviously not perfect, the model developed (as Eq. **[3]**) is a reasonably good fit to the collected data, but it needs to be refined. The model proved to be reasonably accurate when used to predict the MC of the vermiculite required to achieve the desired wood MC **(Fig. 4),** down to about 25 percent. Subsequent analysis of a second set of data showed that the predictions were accurate for these pine samples **(Fig. 5).** This suggests that the model, with differing degrees of refinement, may be relatively robust.

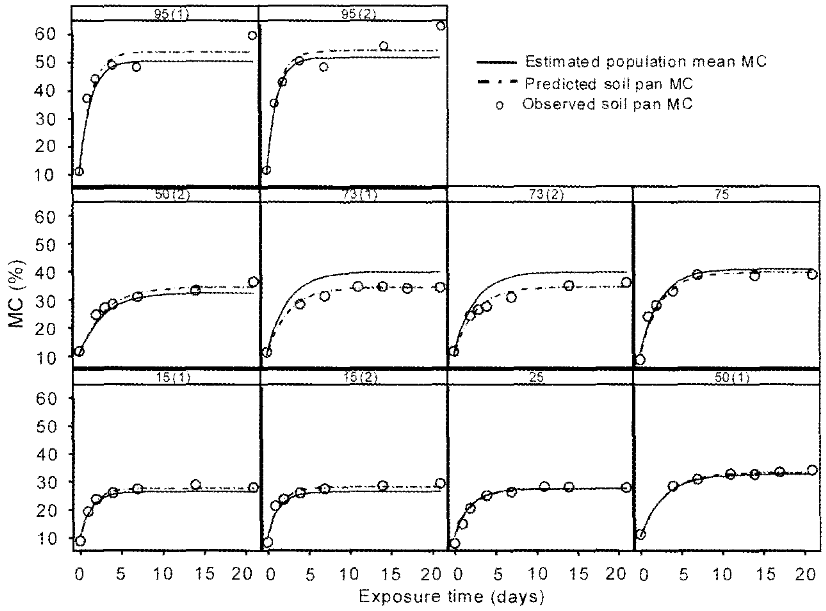

Figure 4. — MC for each vermiculite batch as observed over time (dots). Estimated mean fixed-effects model (Eq. [3]) with random effects set to zero (solid line) and predicted mixed-effects model (Eq. [3]) with estimated random effects (dashed line).

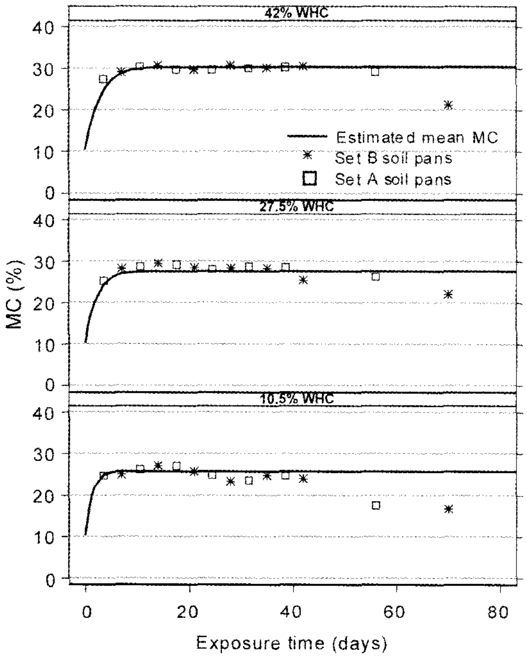

Figure 5. — Measured wood MC from secondary verification phase for three batches of vermiculite (10.5%, 27.5%, and 42% WHC) over time compared to estimated wood MC from mixed-effects model (Eq. [3], with random effects set to zero) independently derived during initial model development.

Conclusions

The MC of southern pine exposed over vermiculite was found to be controllable and predictable by controlling the water-holding capacity of the vermiculite media. This ability to control wood MC may allow us to study the moisture thresholds required for wood decay to initiate over a range of moisture conditions. Dehydration was found to be a problem at low system moisture levels. We found that moisture could be added back to the decay chamber and the moisture environment could be effectively controlled for an extended period. The model developed to relate the WHC of vermiculite media to the eventual wood MC (**Eq. [3]**) was shown to be reasonably accurate. Further experience is needed using different media types or exposure temperatures to assess the broader applicability of the method and models.

Literature cited

American Society for Testing and Materials (ASTM). 1999. Standard method of accelerated laboratory test of natural decay resistance of woods. Standard D 2017-94. Annual Book of ASTM Standards, Vol. 4.10. ASTM, West Conshohocken, PA.

British Standard Institution. 1992. Part 1. Durability of wood and wood based products. Definition of hazard classes of biological attack. BS/EN 335. London, UK.

Cartwright, K. W. Findley, C. Chaplin, and W. Campbell. 1931. The effect of progressive decay by *Trametes serialis* Fr. on the mechanical strength of the wood of Sitka spruce. Forest Prod. Res. Bull. No. 11. British Research Establishment, Garston, Watford, UK.

Cassens, D., B. Johnson, W. Feist, and R. De Groot. 1995. Selection and Use of Preservative-Treated Wood. Forest Prod. Soc., Madison, WI. 104 pp.

Curling, S., C. Clausen, and J. Winandy. 2002a. Experimental method to simulate incipient decay of wood by basidiomycete fungi. Inter. Biodet. Bull. 49:13-19.

__________, __________, and __________. 2002b. Relationships between mechanical properties, weight loss, and chemical composition of wood during incipient brown-rot decay. Forest Prod. J. 52(7/8):34-39.

Kennedy, R. 1958. Strength retention in wood decayed to small weight losses. Forest Prod. J. 8(10):308-314.

Lea, R. 1992. Wood based panel product: Moisture effects and assessing the risk of decay. Information Pap. IP19/92. Building Research Establishment, Garston, Watford, UK.

Mulholland, J. 1954. Changes in weight and strength of Sitka spruce associated with decay by a brown-rot fungus *Poria monticola*. Forest Prod. J. 4(1):410-416.

Pinheiro, J. and D. Bates. 2000. Mixed-Effects Models in S and S-PLUS. Springer-Verlag, New York. 528 pp.

Rapp, A., R.-D. Peek, and M. Sailer. 2000. Modelling the moisture induced risk of decay for treated and untreated wood above ground. Holzforschung 54(2):111-118.

Rayner, A. and L. Boddy. 1988. Fungal Decomposition of Wood. Its Biology and Ecology. Wiley, London.

Sherwood, G. and R. Stroh. 1989. Wood Frame House Construction. Agri. Handbook. 73. USDA Forest Serv., Washington, DC. 260 pp.

Straube, J. 1999. Rain control for walls. Wood Design Focus 10(4):3-12

Viitanen, H. and A. Ritschscoff. 1991. Brown rot decay in wooden constructions. Rept. No. 222. Dept. Forest Prod., Swedish Univ. Agri. Sci., Uppsala, Sweden. 57 pp.

Winandy, J. and J. Morrell. 1993. Relationship between incipient decay, strength, and chemical composition of Douglas-fir heartwood. Wood and Fiber Sci. 25(3):278-288.