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A Nondestructive Longitudinal Laboratory Test Method for Detection of Incipient Ultrastructural Changes in Wood

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Reference

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ABSTRACT

The established methods for determining the effects of ultrastructural changes in structural wood members rely upon traditional, possibly outmoded, testing paradigms. These methods usually involve destructive testing of wood specimens that are exposed to environmental conditions infrequently experienced in buildings. Understanding how the ultrastructure changes with time within the same specimen is crucial for building practitioners in assessing risks to life-safety, assessing remaining service life of structural components, and aiding in the identification of mitigation or remediation measures. The primary agents causing ultrastructural modifications to wood are wood rotting basidiomycetes but may also include other biotic and abiotic agents; the methods contained herein should be applicable to all such longitudinal experiments. This article reviews existing literature on decay testing and validates a new method to assess longitudinal changes in mechanical properties with time using nondestructive test measures at relevant moisture contents. The validation testing shows this method has a good degree of repeatability and should permit the initial detection and monitoring of ultrastructure changes (e.g., decay). The method uses a repeatable, nondestructive four-point testing procedure for specimens controlled to specific moisture contents using energy dissipation as the salient performance metric. Recommendations are provided to refine this novel test method.

Keywords

biodeterioration, decay, wood, fungi, structural testing

Introduction

Wood has been used in buildings for millennia because of its advantageous mechanical properties, availability, and affordability. Structural wood components find widespread use in low- and mid-rise construction, and more recently, in high-rise construction as engineered wood products. With the advent of newer engineered wood products that are finding use in novel building environments, understanding how environmental factors can incrementally impact the structural performance of wood elements is beneficial with respect to evaluations of long-term durability.

One of the main concerns of wood structures is the risk of biodeterioration, which may result in mechanical property losses should these structures be exposed to conducive environmental conditions. The exact cost of premature wood failure to the building industry is unknown but estimates place it at $7 billion for the North American market in the 1990's.[1] The fungi responsible for the majority of these are grouped either as brown or white rots, and form, collectively, the term of wood rotting basidiomycetes (WRBs).

Decay differentiates itself from other fungal attacks in that deterioration occurs in the ligno-cellulose matrix, causing ultrastructural changes affecting macroscopic properties such as stiffness and modulus of rupture (MOR). Ideally, detection of incipient decay, prior to nonrecoverable damage, is preferable to preserve the quality of the structure. Efforts to predict service life of these wood elements have been attempted with varying success and are summarized by Lepage et al.[2]

Traditional methods use destructive tests, which require large numbers of replicates and may have great variability between specimens, exacerbated by the heterogeneity and orthotropic properties of wood. The destructive nature of these tests prohibits the direct measurement of the point of departure in structural properties; it must be inferred by population statistics. Further, to permit comparisons between the experimental variables, prior to the destructive testing, the specimens must be returned to identical testing conditions (e.g., 12 % MC at 20°C), which are not representative of conditions conducive to decay.

Nondestructive test methods minimize experimental errors by permitting reuse of the same specimen in subsequent tests. Incorporation of longitudinal testing parameters, including a range of biotic or abiotic deterioration effects (such as oxidation, thermal degradation, or chemical modification, or biological decay as the concern in this article), will permit the ready incorporation of time factors into the incremental changes in ultrastructure.

Consequently, this article seeks to evaluate the accuracy and utility of a longitudinal nondestructive test method using energy dissipation as an analog for ultrastructural changes in four-point bending at representative moisture contents (MCs). This will provide insight into testing of wood prisms at ranges of MCs, thus permitting treatments to investigate long-term, incremental changes to mechanical properties. The focus of this article is to evaluate a method that detects the impact of decay, but the approach could be extended to any factors that cause incremental changes in the wood cellular structure over time. It will be contrasted with anticipated reductions in decay from contemporary models.

Literature Review

Evaluation of the degree of decay in wood has been of great importance to wood scientists and engineers for many years. Hartley[3] concluded that mass loss was likely the best metric for evaluation of wood decay. This is further elaborated by Wilcox,[4] as other properties such as changes in mechanical properties can be unreliable because of difficulty in measurement of wood strength, type and degree of fungal activity, variations in MC, and spatial/temporal variability of decay. However, from an engineering perspective, the primary concern is change in mechanical properties, specifically as caused by incipient decay, as this results in a loss of structural capacity prior to visible signs of decay.

Reductions in toughness and impact bending are considered the most sensitive to incipient decay, where a 50 % loss in toughness can occur with 1 % weight loss,[5] with reductions of up to 85 % by the time 10 % weight loss was measured. Reductions of mechanical properties in static bending were found to be the next most sensitive to incipient decay, including modulus of elasticity (MOE), MOR, and compression perpendicular to grain. Toole[6] found that 5 % compression had the highest correlation with weight loss.

Further research confirms that incipient decay is characterized by significant decreases in mechanical properties with modest reductions in components and minimal change in appearance,[7] and that incipient decay is best measured by a reduction in strength loss instead of mass loss.[8] The process by which this strength reduction occurs is through depolymerization of hemicelluloses encrusting the cellulose microfibril.

Winandy and Morrell[7] conducted experiments by using four-point bending tests on wood inoculated in accordance with ASTM D2017-05, *Standard Test Method of Accelerated Laboratory Test of Natural Decay Resistance of Woods* (Withdrawn),[9] which uses a soil block inoculum, whereby an inoculated feeder strip is used to decay the specimens. Their approach also included nondestructive test methods, by using sound wave transit time, to estimate the MOE. The four-point bending tests were destructive in nature and the specimens were all dried to 12 % MC before mechanical testing. A four-point test was preferred because it maximized the volume of material under pure flexural stress. As the weakest point of wood governs failure and can vary spatially, maximizing the stressed volume reduces errors associated with spatial uncertainty. Their results showed that strength loss did not occur without weight loss and that the relationship between the two was linear. Degradation of hemicellulose was closely related to wood strength losses.

Subsequently, Curling, Winandy, and Clausen[8] found that strength loss could be measured before significant weight loss by following standard decay tests EN 113, *Wood Preservatives — Test Method for Determining the Protective Effectiveness Against Wood Destroying Basidiomycetes — Determination of the Toxic Values*,[10] and ASTM D2017-05.[9] They found strength loss is a more sensitive measurement of incipient decay than weight loss. They reconfirmed the direct relationship between strength and weight loss.

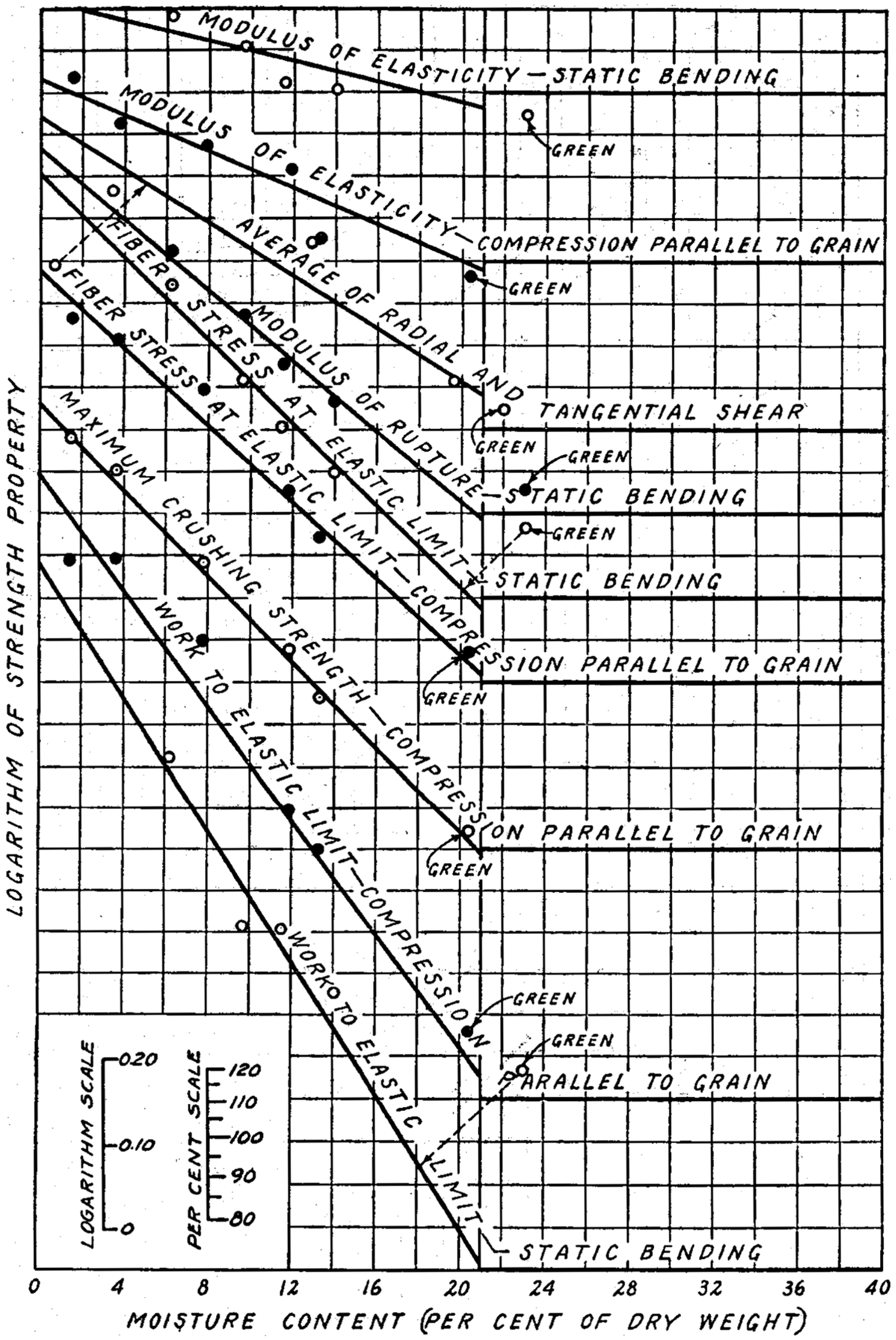
Curling, Clausen, and Winandy[11] further extended the relationship of incipient decay to mechanical loss with the inclusion of chemical analysis of the composition of the wood specimens. Similar to previous work, they used ASTM D2017-05[9] as their inoculation approach and used a four-point bending test to obtain mechanical properties of the specimens. Their findings were that the main chain hemicellulose likely undergoes depolymerization (without dissociation) as part of the incipient decay. The two WRBs used were *Postia placenta* and *Gloeophyllum trabeum*. They found that a 30–40 % difference in mechanical property loss could be attributed to a white rot versus brown rot when used on a softwood. As brown rots preferentially decay softwoods and can be more aggressive, this observation is important for future work dealing with Spruce-Pine-Fir softwood species.

Their results found considerable bending strength loss before significant weight loss, with up to 80 % reduction in work-to-maximum limit (WML) and up to 20 % reduction in MOE before 5 % weight loss was recorded. This was associated with minor reductions in hemicellulose (galactan and arabinan). With the complete degradation of hemicellulose, between 80–95 % of WML was measured and a 20–35 % reduction in MOE, corresponding to weight losses of 5–20 %. A rate of 3.6 % per day reduction in WML was reported. Interestingly, appreciable reduction in MOE only occurred once the cellulose (glucans) were degraded, which suggests the loss of crystallinity in the cellulose is most likely responsible for the stiffness of the wood.

A study on time to initiation of decay in plywood, oriented strand board (OSB), and solid wood was undertaken by Wang et al.[12] Natural inoculation with basidiospores was ensured by providing 1 h of fresh outdoor air, twice daily, to specimens that were kept in chambers with varying relative humidity. The specimens were tested in accordance with the modified ASTM D3043-00, *Standard Test Methods for Structural Panels in Flexure*,[13] outlined by Clark, Symons, and Morris.[14] The specimens were dried to approximately 12 % MC prior to testing. Although this study did four-point flexural testing, the results were not correlated to mass loss, but rather reduction in mechanical properties with environmental conditions.

In all of these tests, the specimens were dried to a reference MC, typically 12 % MC. Testing specimens at different MCs could influence the mechanical properties of concern. The work to proportional limit (WPL) is the deformation energy dissipated up to the transition point from elastic to plastic material behavior. Hoadley[15] suggests it typically occurs around one-half to two-thirds of the maximum stress. The WPL is known to fluctuate with MC; higher MCs have reduced stiffness requiring less energy to cause deformation. Changes in WPL with MC level off at MCs much beyond 20 % MC. The relationship between many of the mechanical properties to MC is shown in figure 1.[16] Tiemann[17] introduced the concept of fiber saturation point (FSP), based on MC below which properties start to change.

FIG. 1 Mechanical properties as a function of moisture content.[17]



All previous tests were destructive in nature, which eliminates the potential to measure the incremental change of decay with time within the same specimen. Nondestructive methods, which remain within the elastic range, may prove beneficial in describing a rate of loss within the same specimen including effects of MC.

Methods

Review of the literature suggests two approaches to optimize the resolution of nondestructive flexural testing. First, four-point bending is preferred as the middle portion of the prism is placed under constant and maximum flexure, compared to three-point bending, where the moment peaks at the center and shear effects could affect results. Further, four-point bending also places a larger volume of the prism under flexure, which is advantageous if attempting to capture phenomena that affect the volume of wood, such as decay. Second, measurement of energy dissipation, in the form of work to ultimate limit or WPL, has a greater resolution to identify incremental changes in structural properties at varying MCs than all other nondestructive methods. The proportional limit was calculated for the first repetition for each specimen at its target MC. The associated displacement at this first repetition was then used as the maximum limit for determination of the work on all subsequent repetitions. Comparisons within specimens over time is thus the amount of work required to achieve the same deflection.

Consequently, the proposed method is to prepare pristine wood prisms, determine their dry mass, equilibrate them to a range of specified MCs, and periodically test them in a nondestructive four-point flexure with load regimes within the elastic range. Once the specimens are at equilibrium conditions, based on ASTM D4933-99R04, *Standard Guide for Moisture Conditioning of Wood and Wood-Based Materials*,[18] the intraspecimen variations between repetitive tests should be negligible. It is anticipated that the only variation in mechanical properties between MC groups would be in general accordance with Tiemann.[17]

The test protocols followed in general conformance with ASTM D4761-19, *Standard Test Methods for Mechanical Properties of Lumber and Wood-Based Structural Materials*. However, to increase the volume placed under pure flexure, a quarter-point four-point test was conducted and at span-to-depth ratios more closely aligned with ASTM D6272-02, *Standard Test Method for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials by Four-Point Bending*,[19] as wood at elevated MCs has features more closely aligned with some polymers with respect to stress relaxation and creep. These standards were preferred over ASTM D3043-00,[13] which focuses on panelized wood products. ASTM D2915-03, *Evaluating Allowable Properties for Grades of Structural Lumber*,[20] was referenced with respect to identification of suitable specimen dimensions.

SPECIMEN PREPARATION

A total of 21 prisms of Lodgepole Pine (*Pinus contorta*) sapwood were cut from the same piece of wood. Lodgepole pine was selected because of its ubiquity in the Spruce-Pine-Fir mix in North America. The prisms were planed to a 12.7 by 12.7 by 101.6 mm in dimension. Prisms were selected to ensure approximate uniformity in grain orientation. A reference corner and face were identified such that the loading was applied perpendicular to the radial axis at consistent loading points. The specimen cross-sectional dimensions were measured at the quarter-point loading points to obtain an average value of the region under flexure. ASTM D4442-07, *Standard Test Methods for Direct Moisture Content Measurement of Wood and Wood-Based Materials* (Superseded),[21] was followed in general conformance in drying the specimens, with the exception of setting the drying temperature to 74°C±2°C instead of 103°C±2°C, to minimize drying damage to wood cellular structures. The number of replicates and their target MC are provided in **Table 1**.

TABLE 1
Specimen description, target moisture content, and replicate number

Moisture Content, %	Specimen ID [Sample Size]
20	5,6,7 [3]
25	1,2, 8,9,10 [5]
30	3,4,11,12,13,14,15,16 [8]
40	17,18,19,20,21 [5]

The specimens were sealed in conical tubes with airtight lids, following the addition of the required quantity of moisture with a pipette. All specimens were maintained at room temperature for 3–5 days to permit absorption of the initial moisture. To estimate the time until full moisture redistribution occurs throughout the cross section of the specimen, a preliminary investigation using the hygrothermal simulation tool WUFI Pro 5, from the Fraunhofer Institute and Oak Ridge National Laboratory, was undertaken. It suggests that the time until full equilibration under tangential flow of a 97.5 % relative humidity environment (equal to approximately 30 % MC for the material file "transverse pine" in the Norwegian University of Science and Technology material database) should take approximately 30 days. The tubes were inverted intermittently, between 7–10 times, to allow distribution of any residual liquid moisture across the entirety of the wood specimens.

APPARATUS

A four-point jig was prepared in general accordance to ASTM D6272-02.[19] However, to ensure test repeatability, three small pins were positioned to ensure the specimen was placed in nearly the exact same location for each test (fig. 2). The base consists of two 25.4-mm (1-in.)-diameter rollers, whereas the top was 12.7-mm (0.5-in.) rollers. Each of these rollers are held supported by a 12.7-mm (0.5-in.) steel plate.

The jig was positioned into an MTI-10K universal testing machine. The machine has an internal displacement resolution of 0.00127 mm, representing an error of 0.0254 % on 5 mm of deflection, neglecting load-cell/jig deformation. Although an linear variable differential transformer (LVDT) to measure center-point deflection would be preferred, as the comparison is based on energy dissipation, the use of the internal displacement measurement was calibrated with an external LVDT. The load cell consists of an Interface 1210AJ-10K, with a 0.02 % full scale output accuracy to 44,545-N maximum load, leading to an error of 0.9 % at a 1,000-N load.

TEST PROTOCOLS

The following parameters were defined for the test protocols, as outlined in Table 2. They are derived from pilot test results outlined in the Results section. The process involves removing the specimens from their containers,

FIG. 2

Repeatable four-point test jig.

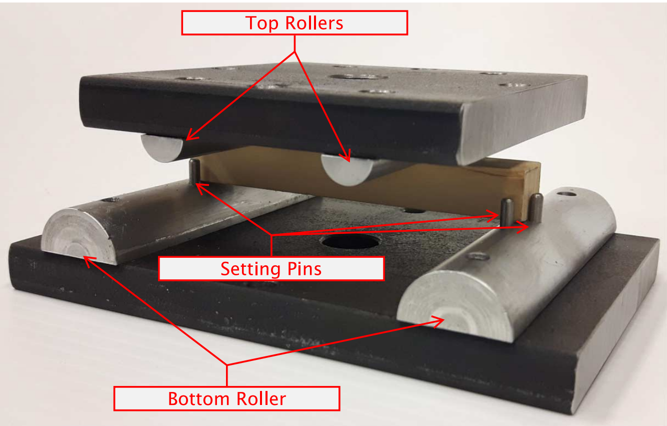


TABLE 2

Loading Parameters

Parameter	Value
Load limit	750 N
Deflection limit	12.7 mm
Loading rate	2.54 mm/min

positioning them against the reference pins in the jig, loading the specimen to the maximum load, unloading, then returning the specimen back into its container.

Upon completion of the test, the specimens were weighed on an electronic scale and distilled water is added with a pipette to achieve the target MC. Postprocessing involves zeroing the results to the nearest data point where the load exceeds 10 N, as this is the minimum load that exceeds the error associated with the load cell. The loads and displacements were converted using the simply supported beam formulae for two equal concentrated loads symmetrically placed at quarter points. Although the conversion to stress and strain is an approximation, provided that all tests are subjected to the same calculation, intertest comparisons remain valid.

Each load-deflection curve is then fitted with a third-order polynomial to ease calculation for differentiation or integration. The area under the curve is integrated to measure the work done to the specimen. The proportional limit was calculated from the first test and converted to a displacement, which was used as the reference point for all calculations of energy dissipation for each specimen.

Results

Validation of the methods first required verification that the underlying assumptions hold. Several pilot studies were first conducted for validation. The main assumptions were as follows:

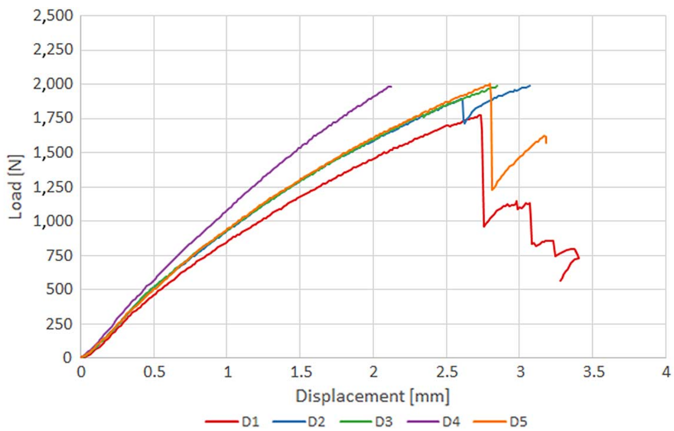
- all stresses remain within the elastic range;
- the within-sample intertest error is insignificant; and
- hysteresis effects are minimal.

These assumptions were tested by completing a destructive test method to identify the elastic range of the specimens.

EVALUATION OF ELASTIC RANGE

Development of the test protocols first required characterization of the anticipated mechanical properties of the prisms, which was completed in a small pilot study. Because the objective is to ensure a nondestructive test, estimates on the proportional limits of wood specimens are required. To evaluate these limits, a series of destructive four-point static flexural bending tests were completed on dry (~12 % MC) replicates derived from the same source material to estimate the proportional limit of the wood prisms (see **fig. 3**). Five replicates were used to evaluate the MOR of the prisms, derived from the same source of *Pinus contorta* wood and placed in the test jig with the applied load in the radial direction.

FIG. 3



Destructive testing of five Lodgepole Pine (*Pinus contorta*) prisms in the four-point flexural jig.

The sample failure load was 1,940±98 N at displacement of 2.6±0.2 mm (mean and 90 % confidence interval). Generally, good agreement among the specimens was observed. These data were fitted to a Ramberg-Osgood model,[22] and the 5 % proportional limit was estimated. The original Ramberg-Osgood model is provided in equation (1), the simplified variables in equation (2), and the 5 % proportional limit in equation (3).[23]

$$\varepsilon = \frac{\sigma}{E} + \beta \cdot \left(\frac{\sigma}{E}\right)^{n} + c \tag{1}$$

$$\delta = \frac{P}{E^{*}} + \beta \cdot \left(\frac{P}{E^{*}}\right)^{n} + c \tag{2}$$

$$P_{prop} = E^{*}\left(\frac{5}{95 \cdot \beta}\right)^{\left(\frac{1}{n-1}\right)} \tag{3}$$

where ε, σ, and E represent the strain, stress, and Young's modulus, respectively, and β, n, and c are fitting coefficients. As the loads are maintained within the elastic range, the conversion from loads and displacements to stress and strain are linear transformations, and so the governing form of the Ramberg-Osgood equation is translated in equation (2) with the applied load, P; displacement, δ; and E^* representing the apparent stiffness. The fitting coefficients and apparent stiffness (E^*) were fitted using a nonlinear least-squares model using the Levenberg-Marquardt fitting algorithm set to a maximum of 100 iterations. The 5 % load to proportional limit, the intersection of the 95th percent MOE with the load-displacement curve, identified as P_{prop}, is determined by equation (3), and the associated proportional displacement may be back-calculated using equation (2).

The proportional limit for the five pilot specimens had a mean of 466 N, with a standard deviation of 187 N. To ensure the proportional limit was captured by a standardized loading scheme, a value of 750 N was selected as the load limit for the experimental protocol as it encompasses 90 % of the proportional loads for all specimens and is less than 40 % of the maximum load.

EXPERIMENTAL RESULTS

The pilot study results informed the testing parameters for determining the WPL of the wood prisms. The prisms were prepared in accordance with the outlined protocols and equilibrated to their target MC. Table 3 provides the timeline for material preparation and testing.

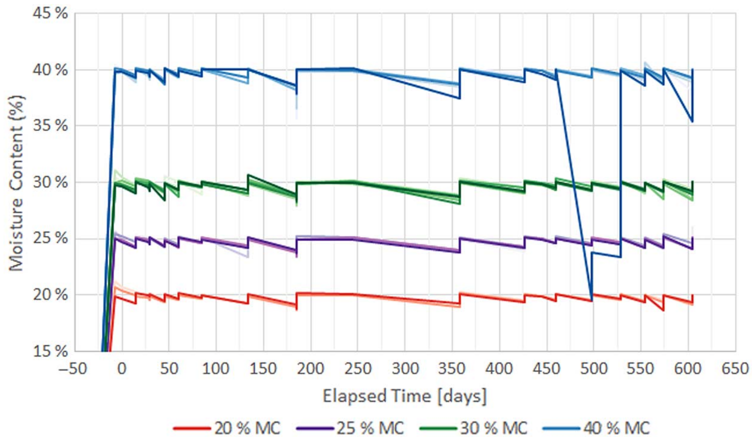
Because of the time for moisture redistribution across the section of the prism, the fourth test (1 month after initial wetting) represents the situation whereby the wood is near full moisture equilibrium. Consequently, all

TABLE 3
Testing timeline

Event	Date	Elapsed Time, days	Notes
Dry mass	2017/04/04	−28	Dried at 70°C
Wetted	2017/04/25	−7	…
First and second rep	2017/05/02	0	…
Third rep	2017/05/16	14	…
Fourth rep	2017/05/30	28	Approx. MC equilibrium
Fifth rep	2017/06/16	45	…
Sixth rep	2017/09/19	140	…
Seventh rep	2018/07/21	445	…
Eighth rep	2018/08/04	459	…
Ninth rep	2018/11/06	553	…
Tenth rep	2018/11/25	572	…
Eleventh rep	2018/12/27	604	…

FIG. 4

Moisture content of all 21 samples over the measurement period. With the exception of specimen 21 (dark blue), all specimens stayed within 1 % of their target MC.



effects refer back to test #4 (1 month after wetting) as the reference baseline. The gravimetrically measured MC of each specimen is plotted in **figure 4**.

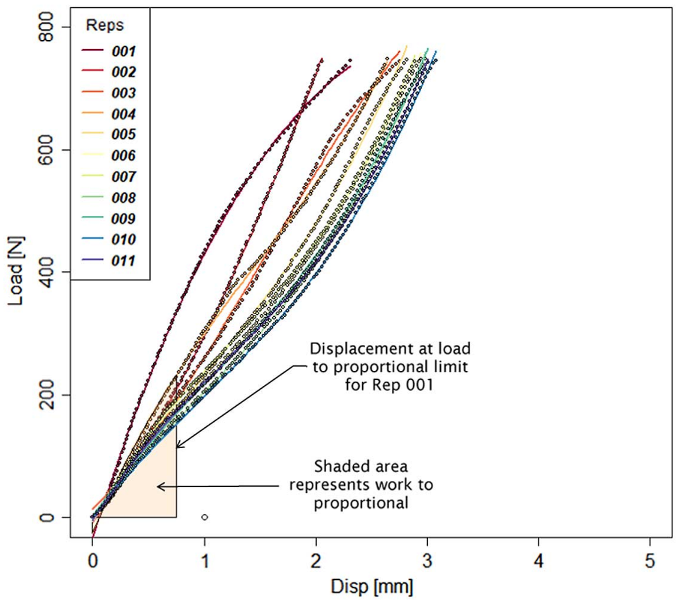
Determining the WPL

The WPL was calculated by first completing a polynomial regression to obtain a best-fitted curve on each load-displacement curve (e.g., each repetition) for each specimen. Integration of the regression line provides the energy dissipation for that specific test at a given deflection. Out of the repeated tests on the same specimen, the cut-off deflection was set to the associated displacement at the load to proportional limit, which was determined for the first test of each specimen at its target equilibrium MC, as outlined in equation (3).

The output for a single specimen C3 held at a 30 % MC is provided as an example in **figure 5**. The measured data from the universal testing machine are shown as filled dots; the third-order polynomial regression is shown

FIG. 5

Repetitions 1 through 11, shaded colors for specimen ID – C3, at 30 % MC. Dots represent measurements, lines represent third-order polynomial regression for each repetition. The shaded region represents the work to proportional limit for rep #4; assumed at moisture equilibrium. Subsequent WPL measurements are made from the displacement at the proportional load limit.



as a solid line; and the color of the dots and lines represent each repeated test ("rep"). The shaded area under the curve represents the WPL for the first test. Comparison of the work among specimens is referenced to this maximum deflection value. The WPL results for each test are plotted in **figure 6**. Individual tests are represented by points, and the group averages are shown by lines; the color represents MC.

In each case, the initial WPL is larger than the subsequent values as deformation energy is dissipated through localized plastic deformation. Further, the initial 30 days also represent the wood prisms achieving moisture equilibrium. Assuming the core is dryer than the exterior, the elevated outer MC of the shell would realize a decrease in the MOE. The amount of energy dissipation for the dryer specimen (MC 20 %) is higher than those nearing the FSP (MC 25 % and 30 %), and significantly higher than the specimens well above the FSP (MC 40 %).

To better show the relative impact of testing with time, the results were normalized to the fourth test, which was estimated to be the point at which the MC within the specimens reaches equilibrium. A linear mixed effects model, with the specimen ID as the random effect, was conducted to estimate the change in WPL as a result of subsequent tests, shown in equation (4).

$$WPL_{rel} \sim Period + 1|Sample\,ID \tag{4}$$

The slopes (*Period*), intercepts (random effect for "1|*SampleID*"), and standard errors are provided in **Table 4**. The results indicate that there is no statistically significant difference between the 20, 25, and 30 % MC specimens, and no difference between the 25, 30, and 40 % MC specimens. A slight difference between the 20 and 40 % specimens was observed, but the effect size is very small. These data are shown visually in **figure 7**, with the grayed area representing the 95 % confidence band.

Relative comparison of the specimens shows that after the span of a year, a difference of less than 7.6 % is expected. This is contrasted with data by Curling, Clausen, and Winandy,[24] which shows a reduction in the work to maximum limit as high as 3.6 % per day for decaying wood. Conversion from WML to WPL suggests

FIG. 6

Energy dissipation with time, colored in accordance with the respective moisture content (red, 20 %; orange, 25 %; teal, 30 %; and blue, 40 % MC).

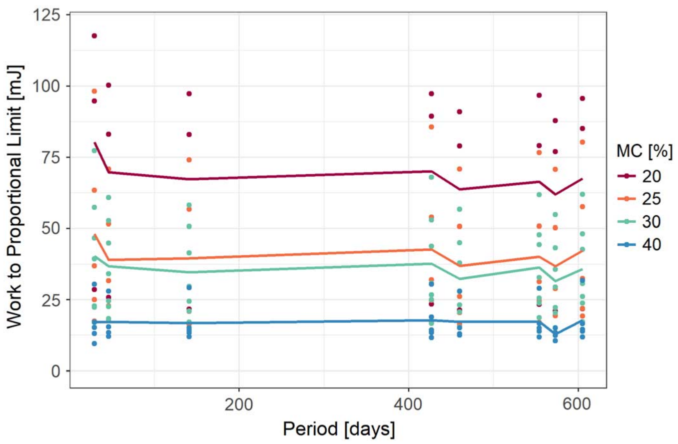


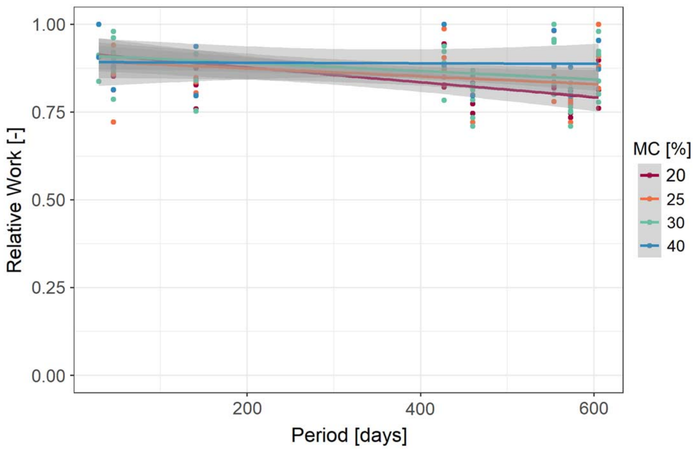
TABLE 4

Linear random effect regression coefficients

Moisture Content, %	Slope	Standard Error	Intercepts
20	−0.00021	0.00005	0.917
25	−0.00011	0.00005	0.897
30	−0.00012	0.00004	0.917
40	−0.00001	0.00007	0.893

FIG. 7

Relative energy dissipation with time, including linear regression lines for specimens at 20, 25, 30, and 40 % MC. The shaded gray fill represents the 95 % confidence band.



a reductions in the range of 40 %.[25] However, even a 1.4 % daily reduction in WPL nonetheless has a signal that is over 70 times stronger than observed in these repeated tests, suggesting that the protocols identified in this article are promising in the detection of incipient decay. When using this test to detect the departure of structural behavior from expected values, a piece-wise regression can be used to detect the inflection point. For added statistical rigor, controls specimens may be included.

Discussion

The standard destructive testing approach toward evaluating incipient ultrastructural changes to mechanical properties of wood specimens requires a large number of replicates and is unable to measure the change in property within the same specimen over time. These limitations add uncertainty to the data collected, as appreciable variation between wood prisms is not uncommon. Furthermore, as most specimens are dried down to a reference MC (e.g., 12 % MC) before destructive testing, the results are not directly comparable to in situ wood elements that are subjected to the biotic and abiotic factors under consideration.

By maintaining loads within the quasi-elastic range and comparing intrasample energy dissipation, a compromise between repeatability and resolution occurs. However, any observed variation must therefore be a result of ultrastructural modifications, assuming a constant MC between the testing points. As a result, the repetitive nondestructive testing (NDT) of four-point static bending of wood prisms appears to be a promising approach toward measuring ultrastructural changes in wood following exposure to factors known to affect structural properties.

Recommendations

Static bending tests on wood prisms at MCs exceeding the FSP demonstrated atypical deformation curves. At the rollers, significant local deformation occurred, in addition to what appears to be shear deflection. The reduction in the shear and flexure modulus at higher MCs suggest that the span ratio originally used may be insufficient. Changing the span to depth ratio, closer to 16:1, as recommended by ASTM D6272-02,[19] should reduce these effects and better capture changes in stiffness of the prisms. Consideration for the decreased load requirements must be made to minimize the trade-off in load-cell accuracy.

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