Modulus of Elasticity Loss as a Rapid Indicator of Rot-fungal Attack on Untreated and Preservative-treated Wood in Laboratory Tests

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The modulus of elasticity (MOE) of wood is a sensitive indicator of rot-fungal attack. To develop an alternative method of rapid assessment of fungal decay in the laboratory, changes in static MOE of untreated and preservative-treated wood were measured during exposure to the brown-rot fungus, *Gloeophyllum trabeum*, and the white-rot fungus, *Trametes versicolor*, in a standard soil bottle assay. Static MOE loss was compared with mass loss. The results showed that the MOE of wood was a sensitive and reliable indicator of rot-fungal attack, regardless of fungus or wood species. The MOE analysis of untreated wood reduced the 12- to 16-week exposure time necessary for the standard mass loss measurement to four weeks. Also, the exposure time for preservative-treated wood was reduced to eight weeks. Untreated wood was determined to be susceptible to decay if the MOE loss was 40% or more after a four-week exposure, while treated wood was considered susceptible to decay if the MOE loss was 40% or more after an eight-week exposure.

Keywords: Fungal decay; Modulus of elasticity (MOE); Mechanical indicators; Preservative treated wood

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INTRODUCTION

Soil block laboratory decay tests are used to evaluate the durability of untreated, modified, or preservative-treated wood to decay fungi in the standards, *e.g.* Chinese standards GB/T 13942.1(2009), LY/T 1283(2011), and AWPA standard M10-12 (2014). These methods evaluate the degree of decay using the percentage of wood mass loss as the indicator. The test is relatively simple, using readily available resources, and provides reasonably good results.

However, the mass loss method usually is not sensitive enough to detect early decay (Nicholas and Crawford 2003). In addition, such methods often require long incubations and a total test time of at least four months from start to finish. Shortening the evaluation time is crucial to the research and development of candidate preservatives or the analysis of new wood modification methods.

The mechanical strength testing of wood can provide quantitative and objective results in determining fungal attack (Hardie 1980; Machek *et al*. 1997; Machek *et al*.1998; Alfredsen *et al*. 2006). Many studies have shown that the mechanical properties of wood, such as modulus of rupture (MOR), modulus of elasticity (MOE), wood toughness, and radial compression strength (RCS) can differ significantly with very minor changes in weight due to decay (Wilcox 1978; Sexton *et al*. 1993; Alfredsen *et al*. 2006).

Toughness is highly sensitive to the early stages of fungal attack; toughness tests are also a simple technique for rapidly assessing the decay potential of a large number of basidiomycetes (Sexton *et al*. 1993). Radial compression strength (RCS) is a more sensitive indicator of initial wood decomposition (< 10% dry weight loss) than weight loss. The RCS of wood can also be used in soil, where the decomposition is limited by low temperature and lack of water or oxygen, or where a rapid estimate of wood decomposition is wanted (Jurgensen *et al*. 2006). Bader *et al*. (2012a,b) studied the fungal decay effect in radial and tangential directions (transverse stiffness) and demonstrated that transverse stiffness properties are more significantly affected by degradation than are longitudinal properties. The reason is that transverse stiffness is mainly effected by the polymer matrix consisting of hemicelluloses and lignin. However, longitudinal stiffness is strongly governed by cellulose micro-fibrils, which are more stable against fungal decay.

It was confirmed that MOE is weakly correlated to hemicellulose content but strongly correlated to cellulose content (Bouslimi *et al*. 2014). Cellulose is depolymerized by brown-rot fungi rapidly during incipient stages of wood colonization and considerable losses in wood strength occur very early in the decay process (Blanchette 2000). Gonzalez and Morrell (2012) also reported that MOE is one of the most sensitive measures of fungal attack. When introduced in pure cultures, decay fungi appeared to be uniformly capable of affecting MOE at very early stages of attack regardless of fungus or wood species, whereas the effects of fungal attack on MOR were less uniform and more variable between wood species. The use of MOE resulted in less variability among samples and appeared to provide a better estimate of preservatives' toxic threshold values (Nicholas and Crawford 2003). Moreover, MOE can be used as a predictor of decay for untreated mini-stakes of Scots pine (Råberg *et al*. 2012).

This research compared changes in static MOE and mass loss of untreated and preservative-treated wood exposed to a brown-rot fungus, *Gloeophyllum trabeum* (Pers. ex Fr.), and a white-rot fungus, *Trametes versicolor* (L. ex Fr.) over the time. The objective of this study was to develop a laboratory method of faster evaluation of the durability of untreated wood and the fungal decay resistance for the preservative-treated wood.

EXPERIMENTAL

Preservative Preparation

The active ingredients of the preservative Copper azole type C (CA-C) and treating solution concentrations are referenced in the AWPA (2013) and Ma *et al*. (2013). The treating solution concentration was 0.278% (0.267% Cu + 0.0107% azole).

Wood Specimen Preparation

Three softwood species, Mason pine (*Pinus massoniana* Lamb.), Radiata pine (*Pinus radiata* D. Don), and southern pine (*Pinus spp.* L.), and two hardwood species, Chinese white poplar (*Populus tomentosa* Duby) and sweetgum (*Liquidambar styraciflua* L.), were used for this study.

The specimens for the decay test were 50 mm by 10 mm by 5 mm (radial by tangential by longitudinal). The number of specimens for each set of variables is summarized in Table 1.

Table 1. Numbers of Wood Specimens Exposed to *Gloeophyllum trabeum* (GT) and *Trametes versicolor* (TV)

Species	The first test*		The second test*				Total
	GT	TV	GT	TV	CA-treated/GT	CA-treated /TV	
Mason pine (*Pinus massoniana*)			20	20	20	20	80
Radiata pine (*P. radiata*)	20	20					40
Southern pine (*P. spp.*)	20	20					40
Chinese white poplar (*Populus tomentosa*)	20	20	20	20	20	20	120
Sweetgum (*Liquidambar styraciflua*)	20	20					40

* The first test was conducted at the U.S.D.A. Forest Products Laboratory, Madison, WI, USA. The second test was conducted at the Research Institute of Wood Industry, Chinese Academy of Forestry, Beijing, China.

Wood Treatment

The specimens were weighed (W_1, g) prior to treatment. The specimens were submerged in the CA-C formulation and then exposed to 10 min vacuum (approx. 100 mm Hg) and a 30-min period at atmospheric pressure to allow any kickback to occur. The specimens were weighed again (W_2, g) after impregnation and wiping. The amount of preservative absorbed by the block (nominal retention) was calculated as Eq.1,

$$\text{Preservative retention (kg/m}^3\text{)} = \frac{(W_2\text{-}W_1) \times \text{Solution concentration} \times 10^{-3}}{0.05 \times 0.01 \times 0.005} \quad (1)$$

where W_1 is the weight of the specimen prior to treatment and W_2 is the weight of the specimen after impregnation and wiping.

The preservative retention is summarized in Table 2.

Table 2. Mean Preservative Retention (Air-Dried Blocks)

Treatment	Retention (kg/m^3)
Untreated	0
Mason pine	2.0 ± 0.19
Chinese white poplar	1.6 ± 0.12

Modified Laboratory Decay Bioassay

The decay bioassay was conducted according to the AWPA laboratory method M10-12 (2014). French square 225-mL (8-oz.) culture bottles with metal screw lids were used for all of the decay bioassays. However, the bottles were laid flat (Fig. 1) because of the longer test blocks. Two decay fungi, a white-rot fungus, *T. versicolor*, and a brown-rot fungus, *G. trabeum*, were used for decay bioassays. One feeder strip was inserted in each culture bottle for each block. The feeder strips were approximately 60 mm by 30 mm by 5 mm and provided a source of actively growing fungal inoculums for the test blocks. The sapwood of southern pine was used to test with *G. trabeum*. The sapwood of sweetgum was used to test with *T. versicolor*.

When the feeder strips in the culture bottles were covered with inoculum, the two test specimens were aseptically placed into one bottle on the top of the feeder strip (Fig. 1). The culture bottles containing the test specimen were incubated at 28 °C with an average relative humidity of 80% to 90%. The exposure time was 0, 2, 4, 8, and 12 weeks. Four specimens were removed from each bottle at each time interval. To maintain the same MC, the test specimens were soaked in deionized water overnight. After the soaking, the specimens were then placed in plastic bags for one day to equilibrate the MC of the specimens. Following this, the MOE value was determined with a mechanical tester (Instron Microtester 5848, Norwood, MA).

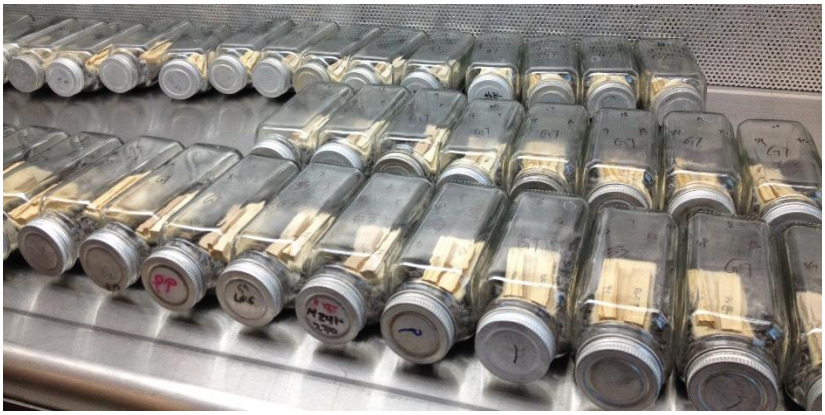

Fig. 1. French square culture bottles

MOE and Mass Loss Measured

Static center-pint bending tests were conducted to determine the modulus of elasticity (MOE) in the test machine with loading and bearing blocks with the radius of curvature of 5 mm. The test span was 36 mm and the loading speed was 1 mm/min. MOE values were recorded for the individual specimens. The percent loss in MOE was calculated for each specimen using Eq. 2,

$$\text{MOE loss (\%)} = \frac{(MOE_0 - MOE_t)}{MOE_0} \times 100 \quad (2)$$

where MOE_0 is the MOE of the specimen without inoculation and MOE_t is the MOE of the specimens after various exposure times.

Before exposure to the fungus, each specimen was weighed after being conditioned (25 °C, average relative humidity about 75%) for seven days ($Mass_0$). After various periods of exposure to the fungus, each specimen was conducted to equilibrium, bending test, oven-dried overnight at 55 °C by turn. Then, the specimen was weighed after seven- days condition ($Mass_t$). The mass loss was calculated using Eq. 3:

$$\text{Mass loss (\%)} = \frac{(Mass_0 - Mass_t)}{Mass_0} \times 100 \quad (3)$$

where $Mass_0$ is the mass of the specimen before rot-fungal exposure and $Mass_t$ is the mass of this specimen after various periods of exposure to the fungus.

RESULTS AND DISCUSSION

MOE and Mass Loss of Untreated Wood

Mass losses and the MOE losses of wood specimens exposed to two decay fungi are shown in Fig. 2. In the second test, when evaluating the MOE and mass loss of preservative-treated wood, untreated wood specimens exposed to the two decay fungi as controls are shown in Fig. 3. Figures 2 and 3 show that, regardless of fungus or wood species, the MOE losses were notably greater and occurred more rapidly than the mass loss trends over the period of rot-fungal exposure. The results further confirmed that MOE loss is a sensitive indicator of rot-fungal attack to untreated wood.

MOE losses of southern pine and Chinese white poplar were severe after two weeks of exposure. The MOE loss of southern pine was 39% and 41%, respectively, when it was exposed to *G. trabeum* and *T. versicolor*. The MOE loss of Chinese white poplar was at least 29% when it was exposed to both *G. trabeum* and *T. versicolor* (Figs. 2 and 3). However, the MOE loss of radiata pine and sweetgum did not show rapid decline. The MOE loss of radiata pine and sweetgum was 8% and 22.7% when exposed to *G. trabeum* and 0% and 9.1% when exposed to *T. versicolor*, respectively. There was no clear relationship between MOE loss and mass loss for radiata pine specimen, which had a 1:1 MOE to mass loss ratio when specimens were exposed to *G. trabeum*. However, southern pine had almost 6:1 ratio when exposed to *G. trabeum* and almost a 105:1 ratio when exposed to *T. versicolor* (Table 3).

Table 3. The ratio of MOE loss/Mass loss of Specimens Exposed to *G. trabeum* (GT) and *T. versicolor* (TV) over Different Time

	GT					TV				
	2 weeks	4 weeks	8 weeks	12 weeks	16 weeks	2 weeks	4 weeks	8 weeks	12 weeks	16 weeks
Radiata pine	0.94	3.05	2.00	1.42		0.00	2.70	4.30	2.94	
Southern pine	5.78	4.92	2.03	1.40		105.13	13.95	4.29	3.42	
Sweetgum	2.65	3.17	1.96	1.99		1.02	2.01	1.79	1.70	
Mason pine	9.49	4.58	2.57	2.24	1.44	6.87	4.71	2.95	2.33	1.33
Chinese white poplar (the first test)	3.17	3.53	2.22	2.02		4.03	2.65	2.24	2.27	
Chinese white poplar (the second test)	3.86	1.84	1.82	1.35	1.18	2.46	1.95	1.75	1.39	1.19

After four weeks of exposure, the MOE loss for all wood species was 40% to 61%, while the mass loss was only 2.9% to 24.8%. In the second test, the MOE losses of Mason pine and Chinese white poplar were as high as 62.5% and 80.5% when exposed to *G. trabeum* and 77.0% and73.0 % when exposed to *T. versicolor*, respectively. The ratio of MOE loss to weight loss of all the wood species were nearly 2:1 to 14:1 (Table 3), which indicates that MOE loss is a more sensitive detector of decay than mass loss. MOE loss can distinctly detect decay at four weeks of exposure to the fungus.

After 8 and 12 weeks of exposure, the MOE and mass losses kept rising, except that the MOE loss of Mason pine had minor fluctuation when exposed to *T. versicolor*.

However, the ratio of MOE loss to weight loss of all wood species declined (Table 3). After 16 weeks' exposure, all the wood had the ratios of about 1:1 of MOE loss to mass loss. This indicated that the MOE loss can serve as an indicator of evaluation the decay lost advantage at late stages of rot-fungal attack. The MOE loss of all test species of untreated wood could be markedly detected after four weeks' exposure. After four weeks of exposure, untreated wood showed at least 40% MOE loss.

Previous reports said that brown-rot fungi are perceived to be more damaging because they are associated with greater strength losses at lower weight losses (< 5% weight loss) than white-rot fungi (Zabel and Morrell 1992). Brown-rot appear to de-polymerize wood in the early decay stages much more rapidly than the decay products can be metabolized (Curling *et al*. 2002). Moreover, many brown-rot fungi use chelator-mediated Fenton reaction in the initial stages of decay as a pretreatment, harboring a novel pathway to improve saccharification yields (Xu and Goodell 2001; Zhang *et al*. 2016). Therefore, brown-rot fungi would be expected to cause drastic strength losses during early decay states (Curling *et al*. 2002; Clausen and Kartal 2003). White rots may completely degrade the wood with weight losses approaching 96% to 97%. White rot fungi can degrade all cell wall components, including lignin, so strength losses are not significant until late stages of decay (Zabel and Morrell 1992). However, this laboratory wood block decay test showed that the MOE of wood was a sensitive indicator of rot-fungal attack, not only for the brown-rot fungus, but also for the white-rot fungus.

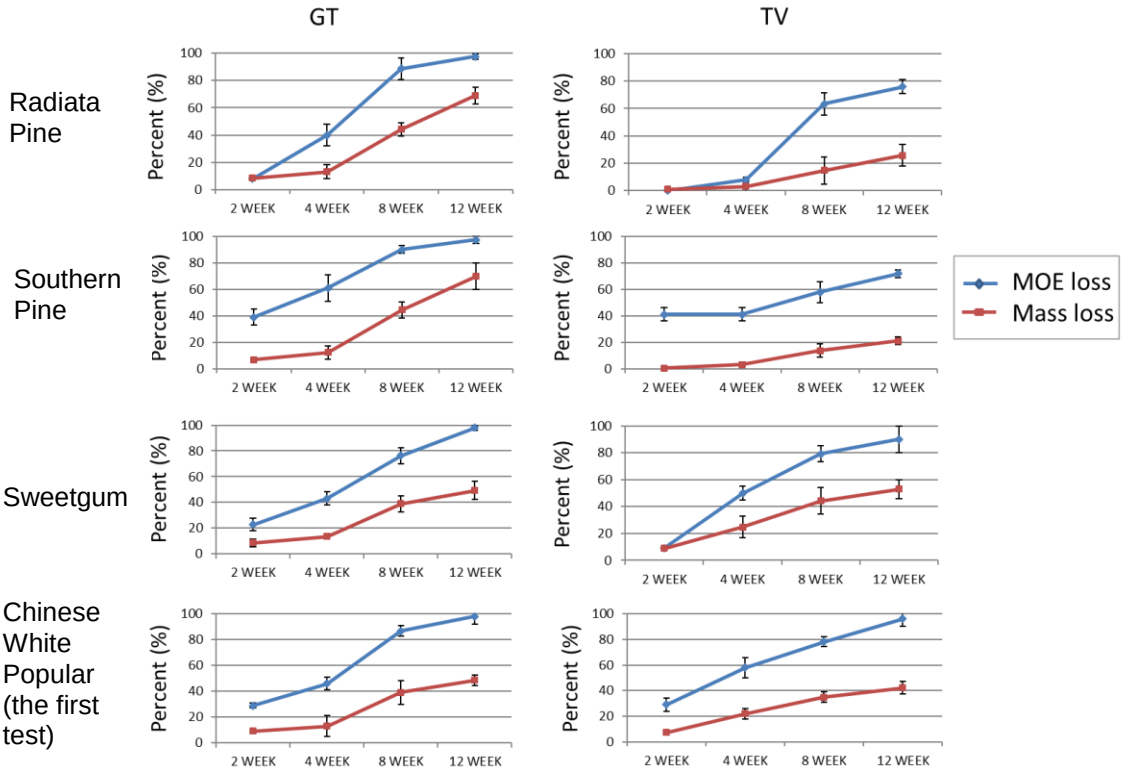

Fig. 2. The MOE and mass loss of untreated wood with different decay exposure time (the first test was conducted at the Forest Products Laboratory, Madison, USA) [GT: *Gloeophyllum trabeum* (Pers. ex Fr.); TV: *Trametes versicolor* (L. ex Fr.)].

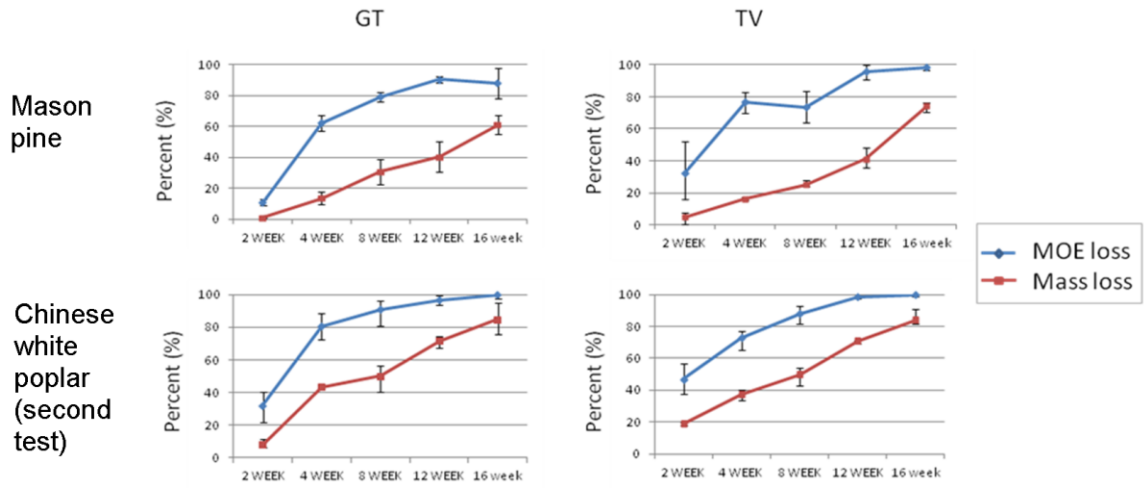

Fig. 3. The MOE and mass loss of the untreated wood with different decay exposure time (the second test at the Research Institute of Wood Industry, Chinese Academy of Forestry, Beijing, China) [GT: *Gloeophyllum trabeum* (Pers. ex Fr.); TV: *Trametes versicolor* (L. ex Fr.)]

MOE and Mass Loss of CA-C Treated Wood

The mass and MOE loss of CA-C treated wood specimens exposed to the two decay fungi are shown in Fig. 4. The controls are shown in Fig. 3.

According to the four-class system of GB/T 13942.1 (2009), the mass loss after 12 weeks of exposure is an indicator of resistance to fungi decay. It is *highly resistant* if the mass loss of wood is less than 10%; *resistant* if the mass loss is more than 10% but less than 24%; and *slightly resistant* if the mass loss is more than 25% but less than 44%. If the mass loss is more than 45%, the wood is *non-resistant* to decay fungi.

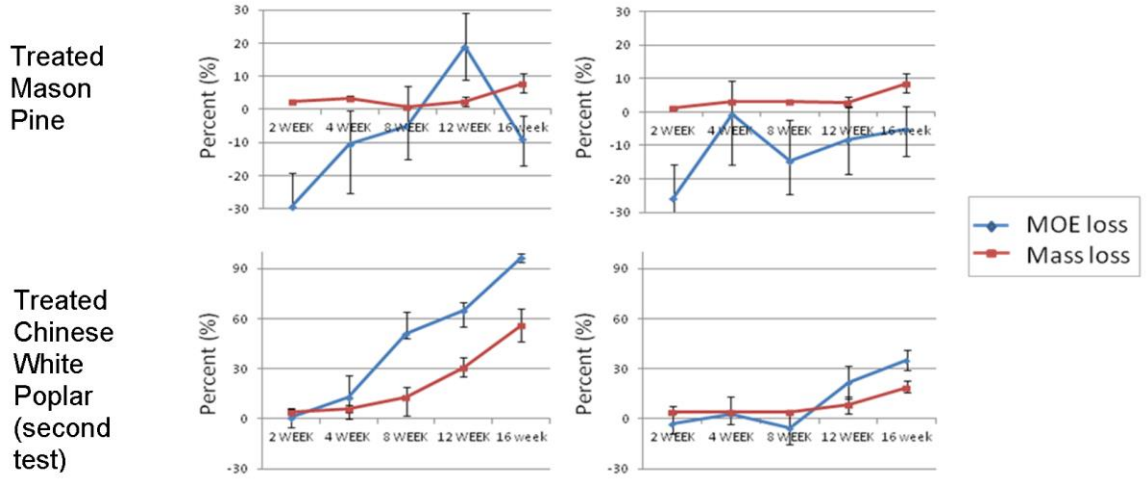

Fig. 4. The MOE and mass loss of the treated wood with different decay exposure time [GT: *Gloeophyllum trabeum* (Pers. ex Fr.); TV: *Trametes versicolor* (L. ex Fr.)]

Mass losses in untreated Mason pine and Chinese white poplar were 40.4% and 71.4%, respectively, following the 12-week exposure to *G. trabeum*, and 41.1% and 90.9 to *T. versicolor* (Fig. 3). The mass loss indicator test results showed that untreated Mason pine and Chinese white poplar are non-resistant against decay fungi. The mass loss of CA-C treated Mason pine was only 2.3% and 2.8%, respectively, following the 12-week

exposure to *G. trabeum* and *T. versicolor*. The results showed that CA-C treated Mason pine was highly resistant to rot-fungal attack. However, the mass loss of CA-C treated Chinese white poplar was 30.5% and 8.2%, respectively, following the 12-week exposure to *G. trabeum* and *T. versicolor* (Fig. 4). These findings showed that CA-C treated Chinese white poplar was resistant to *T. versicolor* attack, but was non-resistant to attack from the brown-rot fungus, *G. trabeum*.

As to MOE indicator, untreated Mason pine and Chinese white poplar had sharp declines of MOE loss over time. The MOE loss of Mason pine was 62.5% and 77.0%, respectively, following a four-week exposure to *G. trabeum* and *T. versicolor*. The MOE loss of Chinese white poplar was 80.5% and 73.0%, respectively (Fig. 3). The ratio of MOE loss to weight loss of untreated Mason pine were about 5:1 and untreated Chinese white poplar were about 2:1, respectively, after four weeks of exposure to *G. trabeum* and *T. versicolor* (Table 3).

The lines of MOE loss of CA-C treated wood over an exposure time were uneven. After four weeks of exposure, the MOE loss of treated Mason pine was negative when exposed to *G. trabeum* and *T. versicolor*. After eight weeks of exposure, treated Mason pine exposed to two decay fungi had no MOE loss. After a four-week exposure, treated Chinese white poplar had minor MOE loss when exposed to *T. versicolor*. After an eight-week exposure, even the MOE loss of treated Chinese white poplar was negative when exposed to *T. versicolor*. As to CA-C treated Chinese white poplar exposed to *G. trabeum*, the MOE loss was 12.8% at four weeks of exposure and rapidly increased to 51.3% at eight weeks. As an analysis of mass loss evaluation, CA-C treated Mason pine was highly resistant to decay fungi *T. versicolor* and *G. trabeum*, while CA-C treated Chinese white poplar only had resistance to *T. versicolor* and was non-resistant to *G. trabeum*. When compared with untreated wood, the MOE loss of preservative-treated wood can detect fungi-decay after 8 weeks of exposure. After an eight-week exposure time, treated wood was highly resistant to decay and had no distinct decline of MOE. However, the MOE loss of treated wood with no resistance to decay was steep and increased to more than 40%. That is, the MOE analysis of preservative treated-wood could reduce the 12- to 16-week exposure necessary for the standard based on mass loss to eight weeks.

Nicholas and Crawford (2003) believed that MOE results appeared to provide a better estimate of toxic threshold values. In this research, preservative treated-wood can be evaluated with a MOE loss indicator after eight weeks of exposure.

Bouslimi *et al*. (2014) found that the weight loss was associated with mannan and xylans losses. Curling *et al*. (2002) reported that early strength loss (up to 40%) of wood during brown decay was associated with the loss of arabinan and galactan components. Subsequent strength loss (greater than 40%) was associated with the loss of the mannan and xylan components. Significant loss of cellulose was detected at greater than 75% mechanical properties loss. In this research, untreated wood after four weeks of exposure and treated wood after eight weeks of exposure to fungi attack were not resistant to decay (MOE loss of at least 40%). Therefore, 40% MOE loss is judged to be the threshold value of early decay.

However, it needs to be emphasized that the current results are providing thresholds for the materials tested in the paper, not necessarily applicable for all other treated and untreated materials. The accelerated laboratory test methods are only the first step to evaluate fungal decay of untreated and preservative-treated wood. It is essential that comparative field tests with known preservatives be included in the development process.

CONCLUSIONS

1. The MOE of wood is a sensitive and reliable indicator of rot-fungal attack, regardless of fungus or wood species.
2. The MOE analysis of untreated wood can shorten the exposure time from 12 to 16 weeks for the standard based on mass loss to four weeks. The MOE analysis of preservative-treated wood reduced the 12 to 16 weeks necessary for the standard based on mass loss to eight weeks.
3. With this laboratory evaluation method, untreated wood at four weeks of exposure and treated wood at eight weeks of exposure to rot-fungal attack were non-resistant to decay if the MOE loss of wood was 40% or more.

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