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Original article

Crack propagation in biodegraded wood

Thibaud SURINI^{1*}, Myriam CHAPLAIN², Patrick CASTERA¹, Gérard VALENTIN²

¹ INRA, Université Bordeaux 1, CNRS, UMR US2B, Bâtiment A11, 351, Cours de la Libération, 33405 Talence Cedex, France ² Université Bordeaux 1, INRA, CNRS, UMR US2B, Bâtiment A11, 351, Cours de la Libération, 33405 Talence Cedex, France

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Abstract

• Fungal attack in wood involves severe mechanical losses, even in the early stages, due to depolymerisation of polysaccharides. The safety of building components could therefore be affected. It is believed that fracture properties could be much more sensitive to decay than conventionally measured properties, such as weight loss.

• In this study, we propose the application of a fracture mechanics test, which measures the fracture toughness, K_{IC} , during the biodegradation process. Two softwoods commonly used in construction, maritime pine and Douglas fir, were inoculated with *Poria placenta*. Samples were removed twice a week to determine the evolution of the decrease in their mechanical properties.

• Toughness was initially greater for maritime pine, but a significant decrease of this property was observed during decay progression, while K_{IC} remained more stable for Douglas fir. For maritime pine there was a loss of 52% of K_{IC} , after 6 weeks of degradation. This species appears to be less durable even with respect to weight loss, which is less sensitive to decay than toughness.

• This is a promising test for bio-damage quantification; however, due to the wood heterogeneity, measurement of the true impact of biodegradation is still difficult.

1. INTRODUCTION

When atmospheric conditions are favourable, commercial timber may be prone to fungal attack. In addition, a wood moisture content of 20% is usually enough to initiate decay (Clausen and Yang, 2007; Zabel and Morrell, 1992), which can have dramatic consequences. It is established that brownrot decays mainly depolymerise polysaccharide components in wood (hemicelluloses, cellulose) (Irbe et al., 2006; Ritschkoff, 1996), which play an important part in wood mechanical resistance (Winandy and Lebow, 2001). Indeed, the degradation of wood polysaccharides and lignin causes a decrease in mechanical resistance, which appears prior to weight loss (Clausen and Kartal, 2003; Przewloka, 2004; Winandy et al., 2000). Moreover, Humar et al. (2006) observed a decrease of modulus of elasticity (MOE) of 7.4% without concomitant loss of mass. Cartwright et al. (1936) concluded that a mass loss of 8.5% of biodegraded ash was associated with a decrease of 15% in compression strength, 26% in bending strength (Modulus of rupture, MOR) and 90% in impact resistance. In general, the modulus of rupture is found to be more sensitive to decay than the modulus of elasticity (MOE) in the early stages (Winandy et al., 2000) of wood decay.

These findings show the necessity of measuring wood decay at its early stage, by choosing a more sensitive criterion. In addition, the mechanical behaviour of biodegraded wood must be understood in order to avoid problems that could appear in situ.

It is known that a 3-point bending test is not appropriate (Winandy and Morrell, 1993), because it does not always measure the true resistance of a wood sample at the biodegraded zone, due to the stress field not being homogeneous along the beam. By contrast, a compression test is sufficient to rapidly detect biodegradation (Gui et al., 1996). However, such a test remains global and is not always representative of a localized biodegradation process, especially in the early stages of the fungal attack.

We propose the application of fracture mechanics tests to measure the resistance of a small area, localized at the crack tip. From these tests, the critical stress intensity factor (or toughness) K_{IC} can be determined. It characterizes the conditions of the unstable propagation of a sharp crack. The experimental determination of this parameter is relatively simple and will be described later. However, its correct determination requires sufficiently large dimensions of the test specimen to remain in the conditions of brittle fracture (Linear elastic fracture mechanics, LEFM) (Bažant and Planas, 1998). If the dimensions are too small, determination of more sophisticated parameters must be made (Morel et al., 2005; Stanzl-Tschegg et al., 1996). Fracture mechanics tests are currently used to characterize wood material: these involve a drying process of natural cracks in wood cell walls that can reduce fracture resistance (Stanzl-Tschegg et al., 1994). Similarly, heat treatments have some negative consequences on fracture toughness, as

^{*} Corresponding author: surini@us2b.pierroton.inra.fr



Figure 1. CT specimen: dimensions and design in TL configuration.

there is a loss of 35% of K_{IC} , after a treatment at 240 °C (Woessner, 2008). Reiterer and Sinn (2002) also reported a severe loss of fracture resistance after heat treatments, whatever the conditions.

As with heat treatment, biodegradation is responsible for the removal of some components of cell walls. It induces the development of clearly visible microcracks, and a decrease of rupture properties. Therefore, the aim of this study is to investigate the relevance of fracture mechanics tests to measure biodamage, and the evolution of toughness properties, according to the time of exposure to decay. As the biodegradation process is highly localized especially in its early stage, small dimensions specimens must be used. The dimensions chosen in the following discussion are slightly small for strict conditions of linear elastic fracture mechanics; however, it is assumed that small variations of toughness can be used to detect and follow biodegradation process.

2. MATERIALS AND METHODS

2.1. Sampling

Two softwood species that are currently used in the building sector in France were investigated. Only heartwood was studied, in order to be representative of timber that is used in wood building. For both species, only one beam, with dimensions $100 \times 100 \times 2000 \text{ mm} (T \times R \times L)$, was used to machine the specimens.

Maritime pine (*Pinus pinaster* Ait.) is a local species that grows in an area of 1 million hectares in the South-West of France. As a consequence, it is of high economical importance. The timber (approximately 30 years old) came from the Landes region and was purchased at the Lesbats sawmill, in Léon. According to the standard NF EN 350-1 (AFNOR, 1994), this species is moderately resistant to decay (class 3). Douglas fir (*Pseudotsuga menziesii* Mirb. (Franco)) is also moderately resistant to decay. It is widely found in French forests, as it is a fast growing species and provides timber with good properties. The timber, (approximately 30 years old) came from the Pomarède sawmill in the center of France.

Compact tension (CT) specimens (Fig. 1) were prepared according to the standard, ASTM-E399 (1990), with dimensions adapted to the biodegradation tests, i.e. $24 \times 24 \times 24$ mm; a midline crack, defined by its length *a* of 8 mm, was sawn along the grain, and completed at the tip with a 1 mm crack using a razor blade (Fig. 1). Two 3 mm diameter holes were drilled on both sides of crack for the load application. Mechanical tests were applied in the tangential longitudinal (TL) plane. In wood, this plane is considered to be more representative of the material, because several rings are simultaneously fractured, representing an average of several years of growth.

For each species and each stage of degradation, 12 replicate samples were prepared from different parts of the same initial beam, in order to limit data variation (Tab. I). This sampling is assumed to limit the scattering of the initial properties of each sample allowing a better comparison at each stage of the period of the fungal attack. In addition, the initial density and the proportion of latewood were used as quantitative descriptors to verify the homogeneity of each sample before the fungal attack. After cutting, samples were conditioned in an oven, at 60 °C, to measure the initial dry mass M_{i60} . They were purposely not desiccated at 103 °C to avoid local deformation and microcracks due to high temperatures. Such a drying procedure would have diminished the natural mechanical resistance of the samples (Stanzl-Tschegg et al., 1994).

After biodegradation and rupture tests, a drying at 103 °C was applied in order to measure the final anhydrous mass Mf_{103} , and calculate the initial anhydrous mass Mi_{103} (Eq. (1)), so that the true weight loss, *WL*, caused by decay, could be obtained (Eq. (2)),

$$Mi_{103} = \frac{Mi_{60}}{(1 + H\%_{60})} \tag{1}$$

$$WL = \frac{(Mi_{103} - Mf_{103})}{Mi_{103}} \times 100$$
 (2)

where Mi_{103} is the initial anhydrous mass at 103 °C; Mi_{60} is the initial mass after desiccation at 60 °C; $H\%_{60}$ is the moisture content of wood at 60 °C; Mf_{103} is the final anhydrous mass at 103 °C.

2.2. Proportion of latewood

The proportion of latewood was measured for each specimen, since it is a descriptive variable of the macroscopic heterogeneity present at the site of the crack. For both species, this determination Table I. Sampling method for each species.



Figure 2. Measurement of crack opening.

was made easy by an abrupt and clearly visible colour transition between earlywood and latewood. This proportion is defined as the ratio between the total width of latewood, visible on the radial tangential (RT) plan of wood samples, and the total width of the specimen. This plan was scanned for each sample and the resulting images were zoomed and analysed using Labview[®] (National Instruments Corporation, Austin, Texas, USA) software. A line was drawn perpendicular to the rings, representing the position of the crack. On this line, the width of each latewood part was visually determined. Finally, in order to calculate the proportion of latewood, the sum of latewood parts was divided by the total number of pixels along this line (representing the total width of the sample).

2.3. Biodegradation tests

The brown-rot fungus, *Poria placenta*, was chosen to degrade samples. This was considered appropriate since brown rots, that attack hemicelluloses and cellulose, preferentially degrade softwoods and are consequently more virulent against these woods (Przewloka, 2004).

A malt-agar medium was prepared at a concentration of 45 g L^{-1} and sterilised for 25 min at 121 °C in an autoclave, according to NF EN 113 (AFNOR, 1996). A 50 mL aliquot of medium was transferred to jars, before adding mycelium. After 2 weeks in a climatic chamber at 20 °C and 65% relative humidity, it covered the entire surface of the medium.

As a fracture test is localized, the degradation should preferentially occur at the site of testing. Thus, some mycelium was delicately laid at the bottom of the crack only, using a razor blade. Wood samples were then transferred to the malt-agar medium. A thin Parafilm band was wrapped around the sides of each sample to prevent fungus from developing on the entire wood surface such that only the crack tip could be attacked, i.e. the site submitted to the highest stresses at later in the testing process.

For each wood species, 12 specimens were removed twice a week for 6 weeks, representing 144 biodegraded specimens (plus 12 control undegraded specimens) for each species. After removal, specimens were sterilised by exposing to UV light and transferred to a climate controlled room with the same air conditions as described above, so that they reached a moisture content of 12%.

2.4. Fracture tests

Before the fracture test, each specimen was weighed and dimensions were determined with a digital calliper, with an accuracy of 10^{-2} mm. The depth of the initial crack was also measured. A Zwick/Roell (Zwick GmbH & Co., Ulm, Germany) machine delivering a maximal force of 500 N was used to exert tension, i.e. mode I of fracture (opening mode). The displacement was regulated at 0.5 mm min⁻¹ resulting in rupture in less than 3 min. This duration is commonly used to avoid viscoelastic effects in mechanical tests on wood.

An extensioneter was also placed on the specimen in order to measure the crack opening during the test (Fig. 2). For simplicity, the critical load, F_c , was taken to be the maximum from the load

displacement curve obtained for each sample. The critical stress intensity factor, K_{IC} (expressed as MPa m^{1/2}), was calculated using equation (3) (ASTM-E399, 1990):

$$K_{IC} = \frac{F_c}{B\sqrt{W}} \times f\left(\frac{a}{W}\right) \tag{3}$$

with

$$f\left(\frac{a}{W}\right) = \frac{\left(2 + \frac{a}{W}\right) \left(0.886 + 4.64\frac{a}{W} - 13.32\left(\frac{a}{W}\right)^2 + 14.72\left(\frac{a}{W}\right)^3 - 5.6\left(\frac{a}{W}\right)^4\right)}{\left(1 - \frac{a}{W}\right)^{3/2}}$$

B and *W* are the thickness and length of the specimen, respectively; whereas *a* is the initial distance of the end of the crack from the load application holes, as shown by Figure 1.

2.5. Statistical analysis

Analysis of variance ANOVA with the risk $\alpha = 5\%$ was performed using Minitab v.15[®] (Minitab Inc., State College, Pennsylvania, USA) software, in order to compare mean values. In addition, a principal component analysis (PCA) was carried out. A PCA enables visually discrimination of individuals (or samples) in a multidimensional space in which the new axes (principal components) are the eigenvectors of the covariance matrix of the initial variables. They are therefore independent of each other. The projection of variables in the plane is represented by vectors with sizes that are proportional to their contribution to this plane. The angle of a variable vector with an axis is proportional to the correlation of the variable with the corresponding component. Such a representation allows the analysis of samples with a complex correlation structure. Statistically a PCA is significant if the first two components represent more than 50% of the overall variability, and the first three around 80% of the variability.

3. RESULTS

With maritime pine, the density was statistically equivalent, regardless of the sample tested (p = 0.62). With Douglas fir, control samples were initially heavier than biodegraded samples, but still were statistically equivalent. The statistical homogeneity for each species at the 95% confidence interval level is presented in Figure 3. We conclude from these results that the evolution of mechanical properties with the progression of decay can be analysed without any significant bias due to material variability.

In order to facilitate the analysis of results and avoid scattering of data, a principal component analysis (PCA) was performed for all measured variables: fracture toughness, density, weight loss, degradation time, and latewood %. PCA takes the eigenvectors of the covariance matrix for these five variables, and provides five principal components that are independent. Figure 4 presents the results obtained in the 1,2 plane (approximately 80% of the overall variability, 53.7% for the first component only). The projection of data in the 1,2 plane (Fig. 4) showed, firstly, that Douglas fir and maritime pine were clearly separated by the proportion of latewood, with mean values of 40% and 34%, respectively. Secondly, the closer position of maritime pine data with respect to the variables indicated that this species had a greater toughness, but also a higher weight



Figure 3. Statistical comparison of the mean initial density of samples (the dotted line represents the global mean; continuous lines represent the number of samples and circles represent mean values for each level of comparison. Mean values that are between continuous lines are equivalent); (a) for maritime pine; (b) for Douglas fir.

loss. Besides, the sensitivity to decay of pine was confirmed by a larger variation of these data according to time of degradation (the progression of decay is indicated by dotted lines and arrows). Finally, the negative correlation between toughness, density and degradation time indicated a strong reduction of toughness with decay.

Table II presents the statistical results (mean \pm SD) for all variables during the 6 weeks of biodegradation. It should be noted that the fracture toughness of both undegraded materials are in agreement with literature values (Boatright and Garrett, 1983; Schniewind and Centeno, 1973). Indeed, the initial value of toughness was 38% higher for pine than for Douglas fir; whereas density was marginally lower for the pine samples. After degradation, the lower durability of pine compared to Douglas fir was confirmed. Measurements of weight loss were used to evaluate this observation further. For each period of degradation, mass loss was lower for Douglas fir than for pine.



Figure 4. Principal component analysis of all measured variables (each point is the mean value of 12 replicates); solid lines indicate the weight of each variable in the normalised 1,2 plane. Dotted lines and arrows indicate the trends of decay progression for both species.

Table II. Statistical values of properties (density, latewood %, weight loss and K_{IC}) after degradation, according to its duration.

	Douglas fir									Maritime pine								
Time of degradation	Density		Latewood (%)		Weight loss (%)		K _{IC} (MPa.m ^{1/2})			Density		Latewood (%)		Weight loss (%)		K _{IC} (MPa.m ^{1/2})		
(days)	Mean	SD	Mean	SD	Mean	SD	Mean	SD	Toughness loss (%)	Mean	SD	Mean	SD	Mean	SD	Mean	SD	Toughness loss (%)
0	0.60	0.034	41.6	5.70	0	0	0.28	0.031	0	0.58	0.031	29.0	6.67	0	0	0.42	0.071	0
4	0.56	0.029	33.9	5.26	0.49	0.15	0.23	0.031	18	0.61	0.027	37.1	6.27	0.43	0.14	0.42	0.053	0
7	0.57	0.017	38.7	4.46	0.22	0.084	0.20	0.046	29	0.62	0.048	37.6	5.14	0.85	0.78	0.34	0.048	19
11	0.58	0.036	39.5	7.01	0.43	0.38	0.24	0.038	14	0.63	0.042	39.2	7.05	0.74	0.67	0.37	0.051	12
14	0.58	0.033	42.1	5.21	0.64	0.59	0.22	0.042	21	0.62	0.066	34.4	9.54	1.95	0.81	0.31	0.066	26
18	0.59	0.033	41.5	7.45	0.95	0.70	0.24	0.041	14	0.60	0.060	29.2	7.39	2.06	1.03	0.32	0.10	24
21	0.55	0.26	37.7	9.48	1.21	0.96	0.25	0.048	11	0.62	0.082	37.0	9.70	2.40	1.31	0.28	0.057	33
25	0.56	0.042	41.9	9.78	2.61	1.94	0.20	0.052	29	0.60	0.057	32.5	7.03	3.78	2.05	0.30	0.072	29
28	0.58	0.031	44.0	5.57	2.00	1.51	0.21	0.034	25	0.60	0.074	33.5	6.55	6.32	4.00	0.25	0.11	40
32	0.58	0.050	43.4	8.57	2.73	2.56	0.22	0.034	21	0.62	0.095	32.8	10.7	5.83	2.82	0.27	0.082	36
35	0.57	0.029	44.8	9.98	2.81	2.62	0.22	0.049	21	0.56	0.071	35.5	9.12	9.63	4.48	0.18	0.080	57
39	0.54	0.023	37.9	3.64	2.67	2.29	0.22	0.032	21	0.57	0.076	33.5	5.56	9.50	5.62	0.21	0.083	50
42	0.55	0.027	35.4	5.20	2.30	1.76	0.24	0.041	14	0.53	0.041	34.7	9.73	10.85	3.02	0.20	0.059	52

For both species, the general trend can be characterized by a simple quadratic fitting, indicating a slow variation at the beginning of the fungal attack (Fig. 5). By contrast, the measurements of toughness revealed a linear fit, as shown by Figures 6a and 6b. This trend, mainly visible with the pine samples, shows a higher sensitivity to measured toughness compared to weight loss, especially at the early stages of degradation. Moreover, a correlation between the duration of degradation and K_{IC} was tested. The degradation was more rapid with pine (Pearson's coefficient = -0.66) than with Douglas fir (Pearson's coefficient = -0.25). The effect of biodegradation was not as clear at the early stages for Douglas fir, even with respect to toughness, K_{IC} .

As Douglas fir was not as degraded as pine the results of weight loss were somewhat hidden by the dispersion of the data. It was also observed that toughness exhibited a small decrease over time of degradation (Fig. 6b). However, toughness values were statistically equivalent (p = 0.23), whatever the degradation, showing that there were no significant decrease in this parameter.

The coefficient of variation (COV) calculated from the standard deviation revealed a high variability of the mechanical property (typically COV exceeds 20), which is characteristic of most strength properties in wood. However, this COV was found to be much higher for weight loss, in excess of 50% in the majority of cases. It shows that mass loss is not a sufficiently reliable criterion.

4. DISCUSSION

We have compared two wood species and found in this study maritime pine to be initially more resistant to crack



Figure 5. Relationship between weight loss and time of degradation; (a) for maritime pine; (b) for Douglas fir.

initiation than Douglas fir, despite its marginally lower density. Taking into account that other mechanical properties of maritime pine are generally also high (MOE = 11300 MPa; MOR = 95 MPa) (Nepveu, 1994), the use of this species for structural applications is appropriate. This is similar to the common use of Douglas fir. One possible reason for the lower fracture toughness of Douglas fir is the strong heterogeneity between earlywood and latewood in this species (density ratio may reach a value of 3), which could be responsible for a non-homogeneous stress state along the crack tip, and possibly higher stress concentration. One disadvantage of the use of maritime pine is its variability in wood properties (Tab. II). This variability has been studied previously (Castéra et al., 1999; Knapic and Pereira, 2005; Machado and Cruz, 2005; Pot et al., 2002) but still needs to be taken into account when designing protocols. In addition, the heterogeneity of wood is responsible for a large variability of toughness. Since fracture tests are destructive, it is difficult to measure the real impact of decay, because the configuration of wood samples (density, growth rings, etc.) can also affect the results.

Despite the drawback of inherent variability, this study reveals a greater sensitivity of toughness measurements over the course of degradation for maritime pine, in comparison with mass loss. Although the variability of samples was minimised by using specimens from the same piece of wood, weight losses were too heterogeneous to draw any conclusions from this data. This may be due to the small size of specimens sub-



Figure 6. Relationship between K_{IC} and time of degradation; (a) for maritime pine; (b) for Douglas fir.

jected to biodegradation tests. The use of fracture mechanics allows the variation of results to be minimised, which is more representative of the true effects of biodegradation. Additionally, in the early stages of degradation, the evolution of K_{IC} is more sensitive than weight loss (Tab. II), proving the value of using fracture mechanics in early detection of decay.

By contrast, it appears that maritime pine is more sensitive to biodegradation than Douglas fir. Whatever the extent of degradation, the loss of mechanical toughness remains below 30% for Douglas fir, whereas it reaches 52% at the end of the test for maritime pine. Furthermore, for pine, the longer the degradation period the higher the decrease of K_{IC} , the correlation of which appears to be linear. Finally, its mass loss continued to increase with time, indicating a higher sensitivity to decay.

Although these two species belong to the same class of natural durability (class 3), there could be an effect of the proportion of latewood, in addition to chemical differences. Indeed, measurements revealed that Douglas fir samples had thicker rings and a higher ratio of latewood than maritime pine samples. As latewood is denser than earlywood due to the thicker cell walls (Donaldson, 1992; Plomion et al., 2001), it can slow down the progression of decay. The state of degradation is consequently less easily determined.

Fracture tests have some drawbacks, because they are difficult to undertake and destructive. But, although these tests were not so relevant with Douglas fir, which was here found more durable than pine, they are still more precise and variations of toughness appear earlier than weight loss, whatever the species. After 6 weeks, the mechanical degradation is not clear enough to be detected on Douglas fir, whereas chemical phenomena may have occurred even earlier than mechanical ones (Clausen and Kartal, 2003). But a longer period of degradation may have justified the utilisation of a fracture test. Indeed, for maritime pine that was more sensitive to decay, it appears in these conditions that fracture mechanical tests can be used in early detection. Besides, these tests also bring some useful information on crack resistance of wood, that could allow to predict the effect of biodegradation on the long-term resistance of structural pieces.

5. CONCLUSION

After a biodegradation process using *P. placenta*, the toughness of maritime pine measurably decreased, even at early degradation, and was more obvious than mass loss. These findings confirm that mechanical consequences of rots are more severe and predictive than weight loss, which is commonly used to determine biodegradation. Douglas fir exhibited lower initial fracture toughness than maritime pine but had a greater stability of this property against decay. These results provide a basis for further work to calibrate biodegradation damage models of wood, but a greater amount of tests and, if possible, tests on larger specimens will be necessary.

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