The application of FT-IR spectroscopy to monitor biodegradation of wood during decay tests

2nd Workshop COST E55 "Modelling of the Performance of Timber Structures"
4-5 October 2007, Eindhoven, The Netherlands.

Nadine Edi Montaruli[1], Elena Conti[2], J.W. van de Kuilen[1], W. Gard[1]

[1] Delft University of Technology, Stevinweg 1, P.O. Box 5048, 2628 CN Delft, The Netherlands, n.e.montaruli@tudelft.nl
[2] CATAS S.p.A, Via Antica 24/3, S.Giovanni al Natisone (UD), Italy, conti@catas.com

Introduction

The study of wood biodeterioration processes plays an important role in the reliability analysis of existing timber structures. When the optimal living conditions for the wood inhabiting micro-organisms occur in a member, the biodeterioration (or decay) process of the material starts. This leads to chemical and physical changes of the substrate, with a consequent reduction of its load carrying capacity.
In order to evaluate the service life of a structure affected by fungi decay, it is important to estimate, besides the strength loss, the rate of decay (or speed) of the process.
Experimental data for the activity, expressed as weight loss in time, of different fungi species on different wood species are reported in studies focused on testing effectiveness of wood preservatives against wood decay according to standards such as EN 113. However, those methods have limitations when used for service life prediction of structures.. The main limits are concerned with the restricted exposure time (generally up to 16 weeks) and the relatively small size respectively volume of the specimen (max 10x25x250 mm^3). The relation between volume of the wood specimen and the rate of degradation has not been reported in the literature so far.
Previous research (Curling et al., 2002) showed that chemical alternations of the cell wall caused by fungi can be related with strength changes. An appropriate method in order to analyse chemical changes is the Fourier transform infrared (FT-IR) spectroscopy, which is a fast and non destructive method.
The present study deals with two aspects:

1. Develop a new protocol for laboratory decay test considering decay value by volume/time relations.

Within the framework of a Short Term Scientific Mission (COST E55) a newly-setup of laboratory decay test has been developed at CATAS Spa Testing Laboratory (Italy).

2. Analysing wood decay by Near Infrared Spectroscopy (NIR)

That research has been conducted at Delft University of Technology (The Netherlands). The Attenuated Total Reflection (ATR) infrared spectroscopy was used to characterize wood decay. NIR infrared spectra of the samples were acquired after different exposure times to the fungus. Spectral differences were analysed by Multivariate analysis (MVA).

Materials and Methods

The timber species used was Norwegian Spruce (*Picea abies*) and Japanese Larch (*Larix kaempferi*) grown in The Netherlands. For each species two sets of 20 specimens differing in dimensions were prepared. The dimension of one set of sample was 10x10x100 mm^3 (ministakes) and for the other 44x44x200 mm^3 (stakes). The edges of each sample were sealed with a waterproof coating in order to prevent the fungi attack starting from the cross sections and proceeding along the longitudinal axis. The samples were oven-dried at 103±2˚C and weighed prior to inoculation.

The test fungi used was the brown rot *Coniophora puteana* (Schumacher ex Fries) Karsten. The inocula was obtained from cultures grown on a malt-agar culture medium.
As culture vessels (incubators) for the ministakes, five sterilized Petri dishes per wood species were used. Each dish was equipped with a basis of moist vermiculite, a plastic mesh and four wood samples, all previously autoclave sterilized. Per sample three inocula were introduced aseptically, so that the mycelium got in contact with the sample surface (Fig. 1). The incubators used for the stakes were autoclavable transparent bags. Sterilized moist vermiculite, plastic mesh and two wood samples were put into each bag. Each sample was inoculated with six inolcula (Fig. 2).

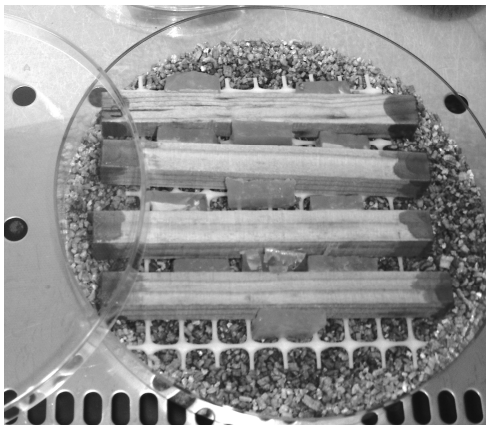

Fig.1 Inoculated ministakes

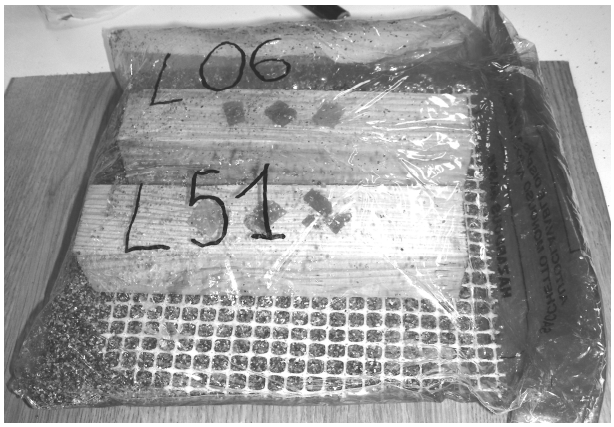

Fig.2 Inoculated stakes

The samples preparation and the inoculation method used in this research aim to simulate fungi attack of timber in reality above ground. In fact, decay problems in buildings are often caused by moisture accumulation, water leakage and water condensation. These generally start from some spots on the surfaces of timber in the structure, and not as a uniform phenomenon of degradation of the whole member.
The exposure time intervals for the ministakes were 2, 4, 8, 12, 16 weeks. The degradation of the stakes is still ongoing at CATAS Spa. The planned exposure times for them are longer than for the ministakes, up to 12 month.. After each exposure time interval, four samples per species have been oven-dried in order to calculate the weight loss percentage relative to the initial oven-dried weight. For the stakes, compression tests will be performed at Delft University laboratory. For the ministakes, weight loss and NIR spectra were acquired after each exposure time.
The FT-NIR spectrometer used is the Spectrum 100 system by Perkin Elmer equipped with a NIRA accessory, ranging from 7800 cm^{-1} to 400 cm^{-1}. With this system, spectra can be obtained on solid wood, without any sample preparation.
Ten NIR spectra were collected from each ministake, so that more than 75% of the whole sample volume was scanned. The mean spectrum for each decayed sample was calculated and Principal Component Analysis (PCA) was performed on the resulting spectral matrix. PCA is a multivariate analysis method that projects the original variables onto a smaller set called Principal Components or PC (Martens et al, 1993). PC scores are the projected locations of each sample onto each PC. Scores can indicate latent structures and clusters of samples.

Preliminary Results

Weight loss
In the considered period of four months for the ministakes and six month for the stakes, the biodegradiation process by brown rot took place successfully. Figure 3 and 4 show the average of the weight loss percentage (ΔW%) for groups of four samples after each exposure time, for the ministakes and the stakes respectively. The weight loss is expressed as the ratio between the difference of the initial weight and the weight after decay over the initial weight, in oven-dry conditions.

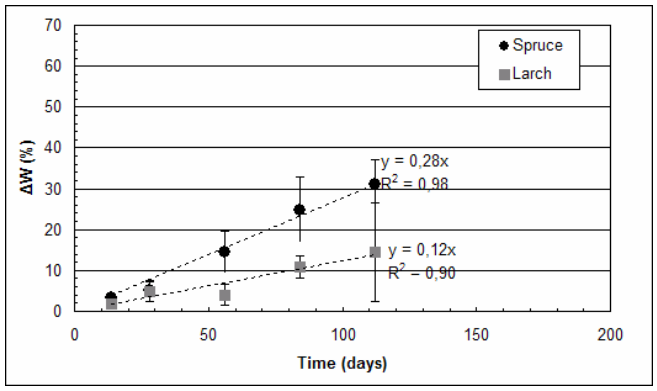

Fig.3 Weight loss in time for ministakes

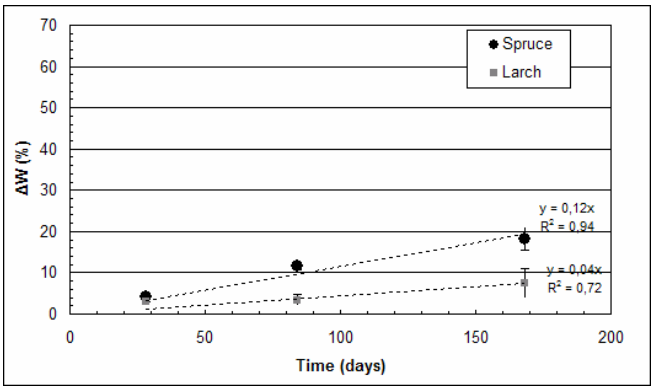

Fig.4 Weight loss in time for stakes

The data demonstrate that Norwegian spruce undertakes a stronger degradation than Japanese larch after the same exposure period, as the weight loss is more than 2 times higher for the spruce ministakes and almost 3 times higher for the stakes. According to the standard EN 350-2 that result was expected since the natural durability class for spruce is 4 (slightly durable) and for Japanese larch is 3-4 (moderately durable).
A more interesting result comes from the comparison between decay process of small and big samples. As expected, the results show that the sample's volume strongly affects the rate of biodegradation. The speed of decay (ΔW%/days) decreases of a factor 2.3 for spruce and a factor 3 for larch, by increasing the volume of 40 times.

NIR analyses
After different exposure times decayed samples showed considerable differences in chemical composition observed by infrared spectroscopy. Figure 5 shows the NIR spectra of spruce ministakes decayed after different incubation time intervals, as well as the reference (sound) sample. Although it is hard to detect differences by visual inspection, it is possible to notice that the absorbance at 4780 cm^{-1} decreases as the incubation time increases. This peak is associated to the cellulose degradation (Kelley et al, 2002).

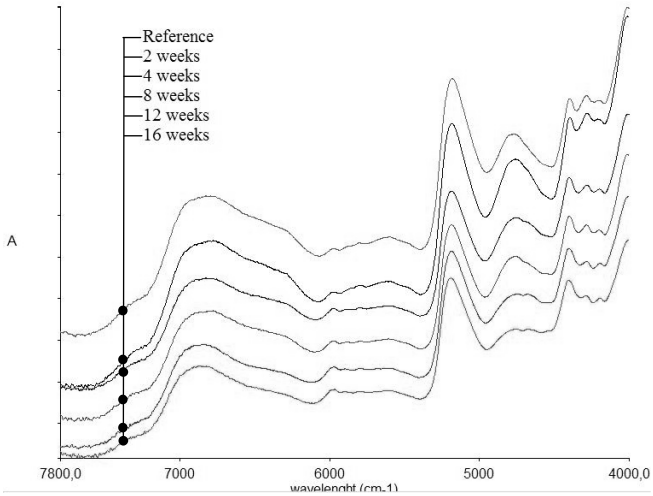

Fig.5 NIR spectra of decayed spruce

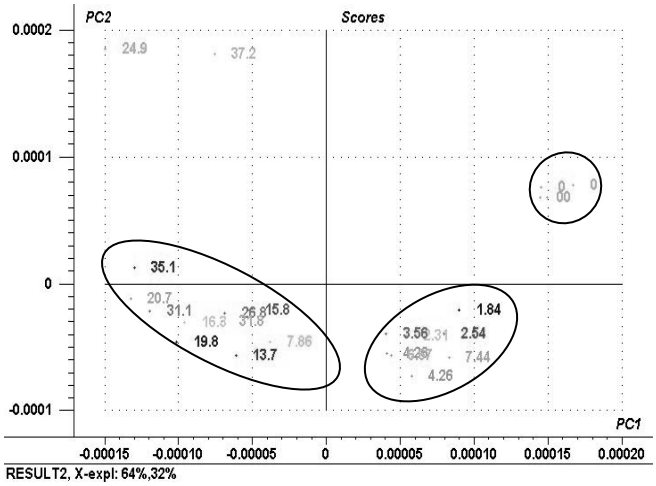

Fig.6 PC analysis discriminating decayed spruce

Figure 6 shows the score plot for the two first principal components which describe the 96% of the total variance of the spectral data. Each score represents a sample, based on its chemical (spectral) constituents. The samples in the score plot are "labelled" with their measured weight losses. The graph shows clearly three clusters, representing sound samples (ΔW=0), low (ΔW<10%) and high

(ΔW>10%) decayed samples (from the right to the left side of the PC1 axis). When the samples are labelled by their exposure time t, the three clusters in Fig.6 represent sound samples (t=0), low (t<1 month) and high (t >1 month) decayed samples. Some outliers are present in the score plot as well. These are situated in the upper left part of the plot. Although the outliers are characterized by high weight loss, their chemical changes are different from the other highly decayed samples. They will be further investigated. It seems to be possible that those samples will show a stronger link with clusters in terms of strength losses.

Conclusions

A new test decay procedure has been developed in order to induce decay in two sets of samples differing in volume sizes. It was proven that the rate of degradation is strongly influenced by the volume of the samples: decreasing up to one third when increasing specimen's volume from 10^4 mm^3 to 40 times bigger for larch samples.
FT-NIT technique and multivariate analysis were applied to characterize wood decay. The spectral analysis showed that it is possible to discriminate sound and decayed samples by infrared spectroscopy.
Within the decayed samples, it was possible to discriminate between "low" and "high" decay.
Due to these preliminary results it is expected that by increasing the number of samples and the incubation time intervals, more clusters (levels of decay) can be obtained with IR spectroscopy and MVA.

References

Curling, S., C. A. Clausen, et al. (2002): Experimental method to quantify progressive stages of decay of wood by basidiomycete fungi. International Biodeterioratian & Biodegradation 49: 13-19.
Martens, H. and T. Næs (1993): Multivariate Calibration. Chichester, John Wiley & Sons.
Kelley, S. S., J. Jellison, et al. (2002): Use of NIR and pyrolysis-MBMS coupled with multivariate analysis for detecting the chemical changes associated with brown-rot biodegradation of spruce wood. FEMS Microbiology Letters 209: 107-111.