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Section 2 **Test Methodology and Assessment**

Weather degradation of thin wood samples

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ABSTRACT

Untreated wooden surfaces degrade when exposed to varying doses of natural weathering. In this study, thin wood samples were studied for weathering effects with the aim of modeling the degradation utilizing Near-infrared hyperspectral imaging. Several sets of samples were exposed outdoors for time intervals from 0 to 21 days, and one set of samples was exposed to UV-radiation in a laboratory chamber. Spectra of earlywood and latewood were extracted from the hyperspectral image cubes and changes in the spectra were modeled as a function of UV solar radiation to see if the weathering deterioration was reflected in the NIR spectra. The model was obtained using Tikhonov regression, an algorithm that yields robust prediction models when predicting new test data. Lignin and holocellulose content were estimated on selected samples separately for early- and latewood using a thermogravimetric analysis (TGA). The thermogravimetric curves showed a clear trend with the progress of weathering of the samples, for both earlywood and latewood and both for the outdoor and the UV chamber exposed samples. This indicates that NIR spectroscopy can also be used to model lignin content in the wood. Further studies are planned to confirm this.
The result from this work is a first step towards a weather dose model determined by temperature and moisture content on the wooden surface in addition to the solar UV radiation.

Keywords: degradation kinetics, hyperspectral imaging, UV radiation, regression models, lignin

1. INTRODUCTION

The use of wood outdoors is widespread in various applications, one of them being as claddings on building façades. There is a trend moving towards less surface treatment of the wood on building façades to obtain a naturally weathered result. However, surfaces of the wooden elements are vulnerable parts of the structures and during service life they are exposed to various mechanical, environmental and biological agents causing ageing, weathering and decay processes. To ensure optimal choices of wood species there is an increasing demand for a thorough understanding of the deterioration mechanisms of wood during outdoor exposure. While it is known that the characteristic grey patina visible after a few months of exposures is mostly caused by photodegradation of lignin in middle lamella by UV radiation (Williams, 2005), there are several other factors involved, such as moisture, temperature, biological growth, chemicals and mechanical abrasion (Gobakken, 2009). The elements in façades made of wood can often cause non-uniform degradation patterns such as shown in Fig. 1. Various architectonical solutions causing geometrical patterns and shapes are generally the main reasons for the heterogeneous appearance since these affect the microclimate on the surface. The microclimate on a wooden façade differs from the exterior climate and can be modelled using ray tracing to account for micro scale variations of the solar irradiance, temperature and moisture on the wall, as presented in Thiis et al. (2015). In order to foresee the degradation on the different parts of a wall, accurate prediction models of wood degradation as a function of the main climatic factors are crucial.

The goal of the present work was to assess the degradation of wooden surfaces caused by weathering and to model its kinetics by means of near infrared (NIR) hyperspectral imaging. Previous studies have shown that NIR spectroscopy and hyperspectral imaging are well suited scientific tools for rapid and non-destructive characterization of wood surfaces (Sandak et al. 2016a, Agresti et al. 2013). One sample-set of thin wood was exposed outdoors and one sample-set was exposed in a UV radiation chamber, and the study has been focused on the influence of UV radiation on the weathering of the thin wood and its perceptibility in NIR spectral images of the exposed wood samples. According to Grossman (1994), a widely used practice has been to use total energy from the integrated solar spectrum as the timing variable for the amount of radiation a sample has been exposed to. This is obtained by integrating over the entire spectral power curve of sunlight, and multiplying by exposure time (Williams, 2005). Modeling the degradation as a function of UV radiation using multivariate regression techniques was therefore carried out to explore how the deterioration of the early- and latewood can be predicted and to define a weather dose for the degradation.

Figure 1: Unevenly exposed façade in Oslo, Norway.

2. EXPERIMENTAL METHODS

2.1 Sample preparation and exposure

Experimental samples were prepared from one piece of Norway spruce wood (*Picea abies*) on a slicing planner (Marunaka) to a thickness of ~100µm and an exposed surface of 30mm x 35mm.

A total of 105 samples were exposed outdoors in Ås, Norway, facing South at 45 degrees as shown in Fig. 2. On the first day 21 samples were put outside for exposure, and then one sample was collected each day and stored at room temperature. After 7 days a new set of 21 samples were exposed, and the following days one sample was collected each day following the same procedure as for sample set 1. This procedure was followed for 5 sample sets of 21 samples each. After collecting all samples, they were stored in darkness in a climatic chamber with a constant temperature of 20°C and 65% relative humidity to obtain an acclimatized weight before further processing. Weather data for the location was obtained from a national weather station located approximately 200m away from the exposure site. The weather station has a pyranometer that measures the solar UV radiation in the wavelength region (298 – 385) nm, which was used as response values when modelling the wood degradation from the NIR spectra.
A supplementary set of 30 samples were exposed in a UV chamber of type Atlas UVTest^{TM}. The Atlas UVTest^{TM} has eight UVA-340 lamps installed with an output of 0.89 W/m^2/nm each. The samples were exposed in the UV chamber for 1 to 10 cycles of 2.5h continuous UV radiation followed by 0.5h water spraying.

Figure 2: Thin samples exposed outdoors facing south.

2.1 Thermogravimetric analysis

Thermogravimetric analysis was carried out for earlywood and latewood separately for selected samples, both from the ones exposed outdoors and from the ones exposed to UV radiation in a chamber. The samples were cut using a scalpel to separate earlywood and latewood. The theromgravimetric analysis was performed using a Simultaneous Thermal Analyzer coupled with a Fourier Transform InfraRed spectrometer (STA FF9 F1 Jupiter, NETSCH, Germany) to estimate the lignin and holocellulose (cellulose + hemicellulose) content. The pieces of cut samples of earlywood and latewood in the containers for the STA are shown in Fig. 3. The samples on which this additional measurement was performed had all been placed outside on the same day, and the samples were chosen such that the whole range from 1 to 21 days of exposure were covered, with equal intervals.

Figure 3: The samples cut into pieces of earlywood and latewood in the containers prior to the chemical analysis.

2.2 Spectral imaging

The spectral imaging measurements were conducted in a laboratory setup with a pushbroom-type hyperspectral camera (Specim, Oulu Finland), which has a Mercury Cadmium Telluride (MCT) detector, sensitive in the near infrared region (1000 – 2500 nm) distributed over 256 channels. One dimension of the detector is used for the spectral separation and the other for imaging one of the two spatial directions so that one line is recorded each time with a spectrum in each pixel. The second spatial dimension is obtained by moving the camera over the sample using a translation stage. A schematic drawing of the pushbroom principle is shown in Fig. 4. On average it takes 5 seconds to scan the sample. The spatial resolution of the setup was approximately 100 µm. The hyperspectral image acquisition was carried out in transmission mode using a custom setup; backside illumination with halogen lamps below a semi opaque glass plate and another transparent glass plate transmitting NIR radiation above the samples. At the end of the scan of each sample, a short image with the shutter closed is carried out and the mean signal from each line in this dark image is subtracted from each band in the hypercube. This procedure is performed to remove the bias level and to correct for pixel-to-pixel variations in the detector. A white calibration has to be performed in order to remove the spectral signal from the lamps. However, a regular white calibration spectralon plate cannot be used in transmission mode. To obtain the white calibration, an image of the empty glass plate was therefore used so that all the sample images were divided by this image after subtraction of the dark signal. The dark subtraction and the white calibration can be formulated mathematically as in Equation 1.

$$I_{corr}^{i,j,k} = (I_{raw}^{i,j,k} - \overline{D}^{i,k})/(W_{raw}^{i,j,k} - \overline{D}^{i,k}) \qquad (1)$$

where I is the image frame, D is the dark image and W is the white calibration image. Indices (i, j) and k correspond to the pixel and channel numbers, respectively.

The earlywood and latewood parts can be identified on the hyperspectral images. In order to automatically extract spectra from the early- and latewood separately, a masking based on Principal Component Analysis (PCA) was applied to the images. The recorded transmission spectra were transformed to absorbance using a log10 transformation procedure. The ten first and ten last bands were excluded from the spectra, since they were entirely dominated by noise. Since various instrumental influences perturb the actual signal, the spectra were smoothed before the analysis using the least squares based smoothing algorithm developed by Savitzky & Golay. The advantage of using this method rather than a moving average is its ability to smooth the spectra without the undesired effect of also smoothing over any peaks in the spectra and reducing their intensity, which may amount to removing important information. Finally, Extended Multiplicative Signal Correction (EMSC) was carried out on the spectra to separate variations due to physical phenomena e.g., light scattering from the chemical variations we wish to model (Martens et al 2003). The basic idea of Multiplicative Signal Correction (MSC) is to fit a regression model of all the spectra in an image to an ideal (reference) spectrum and a flat baseline, and then use the coefficients to perform the correction. EMSC is an extension of MSC that includes terms that account for variation such as differences in scattering over different wavelengths (Kohler et al., 2009). The EMSC correction was performed for all the spectra in all the samples using the HySpec Toolbox (http://nofimaspectroscopy.org)

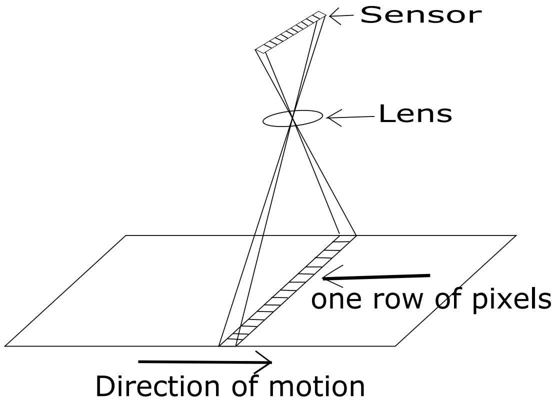


Figure 4: Principle of the pushbroom technique were one line is imaged and the sample is moving so that one line is imaged for each position, which will yield the full image.

2.2 Regression models

After pre-processing was completed, the data ready for analysis consisted of four data sets which were the earlywood and latewood mean spectra for each sample in each group (outside exposure group and UV-chamber group. Regression analysis was carried out on the mean spectra of early and latewood separately as predictor variables and total cumulated UV solar radiation for each sample as response variable. The set of predictor variables and the response variable are all continuous, and so the point of departure is Ordinary Least Squares Regression, of the familiar form: $y = \beta_0 + \beta_1 x_1 + \ldots + \beta_n x_n + \varepsilon$. Here β_0 is the expected value when all $x = 0$, $\beta_1, \ldots, \beta_n$ are the coefficients associated with each variable, and ε is the error term representing variation not accounted for by the model. In linear algebra notation the terms can be arranged as:

$$\mathbf{X}\boldsymbol{\beta} = \mathbf{y} \quad (3)$$

where the rows of **X** correspond to observations and the columns correspond to variables, with a

vector of ones added to account for the constant term. **β** is a vector containing the regression coefficients and **y** is the response vector. The aim of the regression is to derive a model that predicts new observations accurately and reliably, given an observation with response y from a population with an unknown mean μ, an associated explanatory variable x, and an estimate of y given by some function $\hat{y} = f(x)$. A challenge with all regression models is the trade-off between increasing the model complexity or the model robustness. A model with increased complexity may predict well for the set on which it was trained, but it might not generalize well to new data. By relaxing the requirement that the model should be completely unbiased to the training data, it is possible to obtain a model that has stronger predictive power. The regression method chosen in this study named Tikhonov (Kalivas 2012) regularization has a regularization term added to the least squares estimator in order to increase the model robustness.
To check the validity of the models, the datasets were split in two, with 2/3 of each dataset assigned as training data and the remaining 1/3 as test data. The observations were assigned at random, and the model fitted to the training set was used to predict for the test set.

3. RESULTS AND DISCUSSION

The appearance of the wood samples changed after only one day of outdoor exposure of natural weathering. Fig.5 shows four of the samples exposed for 0, 2, 5 and 20 days. It can be clearly seen that the colour turns more yellow (confirmed by additional CIE L*a*b* measurement) and fibres were consequently removed from the surface with the progress of degradation. The drastic change to the chemical structure of the measured samples already after one day of exposure was more dominant for the zone of early wood, as reported in Sandak (2016b). Erosion rate has been previously investigated by several authors (Williams et al. 2001a,b,c, Sandberg 2005) however regarding longer periods of time. It has been reported that during the first several years of natural weathering, early wood eroded much more quickly than late wood (Williams 2001b).

Figure 5: Samples from different sample sets exposed for 0, 2, 5 and 20 days (from left to right).

After preprocessing of the NIR spectra it was noted that parts of the spectra had a very distinct feature at 1850 nm. The spectra were captured from samples that had been stored in a separate envelope. It was concluded that this feature was due to different moisture conditions in the storage of the samples. This had affected the spectra of the samples since the samples are very thin and very sensitive to climate conditions. These samples were therefore excluded from the regression analysis. In spite of the diminished number of samples, the regression results of the mean spectra for early and latewood of the outdoor exposed samples as a function of total UV radiation (showed in Fig. 6) yielded at good R^2 = 0.92 and R^2 = 0.87 on the validation set for early and latewood respectively. The corresponding results for the samples exposed in the UV chamber are shown in Fig. 7. Also here there are good regression performances with R^2 = 0.79 and R^2 = 0.91 on the validation set for early and latewood respectively. This means that NIR spectroscopy can be used as an indicator on the weather deterioration of the wood surface. Moreover, the hyperspectral imaging yields the spatial information in addition to the spectra, so that the models could be obtained separately for early and latewood. However, it should be noted that UV radiation alone is not enough to predict weathering degradation since it is known that the effect of a certain amount of UV energy will also depend on the moisture and temperature conditions of the surface (Grossman 1994). This could also be confirmed by a poor fit when the model calibrated on the outdoor exposed samples was applied to predict the UV chamber exposed samples.

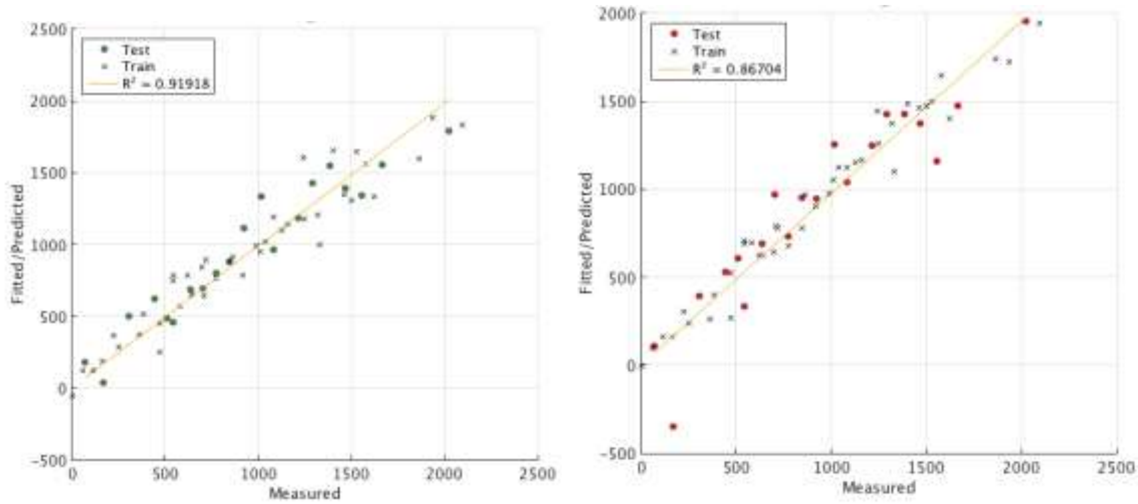


Figure 6: Prediction versus measurements of UV-exposure on earlywood (left) and latewood (right) spectra from the outdoor exposed wood samples. The cross points (67 % of the data set) were used to train the model and the circles (33 %) were used as independent validation sets.

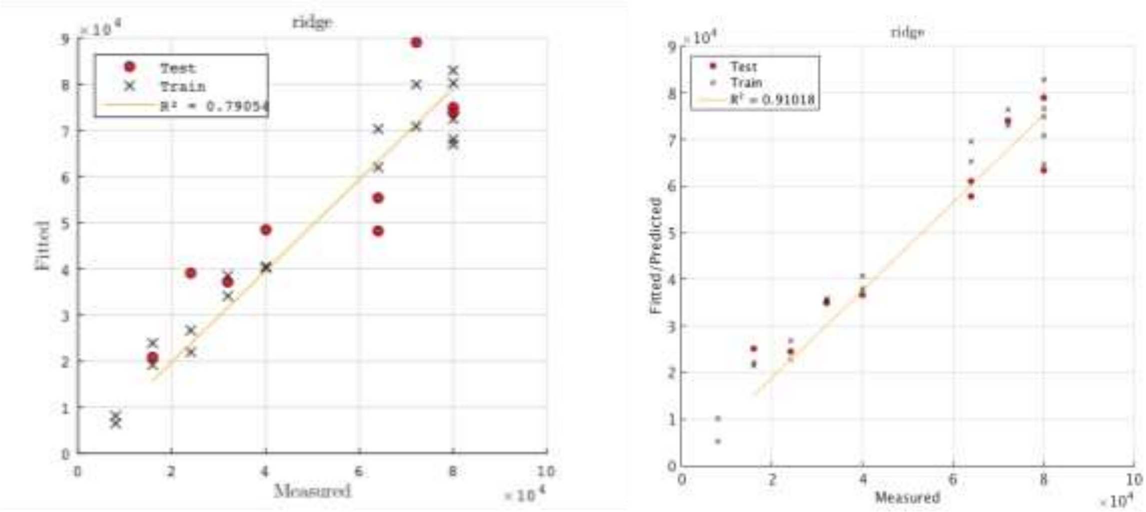
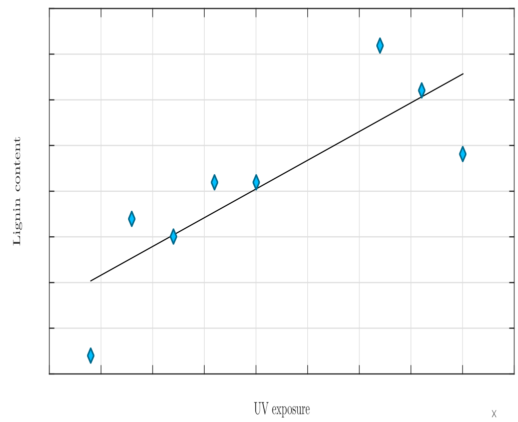


Figure 7: Prediction of UV-exposure on earlywood (left) and latewood (right) on spectra from outdoor exposed wood samples. The cross points (67 % of the data set) were used to train the model and the circles (33 %) were used as independent validation sets.

The thermogravimetric analysis can be used to estimate the lignin content relative to the total weight of the sample. The estimates of lignin content for earlywood for the outdoor and UV chamber exposed samples are plotted as a function of the amount of UV radiation in Fig. 8. Note however that the values are relative to the weight of the sample, and the weight changes with the amount of weathering. Moreover, the number of points in each group is small and the spread in the values is large. In order to confirm whenever there is a trend in the thermogravimetric analysis with increased weathering, the thermogravimetric curves were studied and the mass loss values were interpolated for all degrees in the temperature interval and the mass loss relative to the mass loss of sample 1 (exposed for 1 day) was computed for all the samples. The first derivative of these curves is shown in Fig. 9 to visualize how the mass loss and the differences in the temperature values for the peak of the mass loss for the different UV exposures. The figures display the region from 250 °C to 400 °C, Nitrogen was used to heat the samples up to 360°C, then Oxygen was applied to burn the rest of the sample. From the curves it can be seen that there is a clear trend both in amplitude and temperature value with the amount of UV exposure, both for earlywood and latewood, for outdoor exposed samples and UV chamber exposed samples.



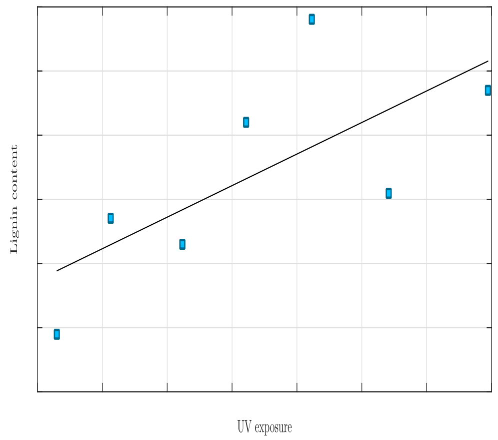


Figure 8: Estimated lignin content relative to the weight of the samples as a function of UV exposure for earlywood exposed outdoors (left) and in UV chamber (right).

4. CONCLUSION

Thin samples exposed outdoors or to UV radiation showed visible degradation after very short exposure times, both for samples exposed outdoors and samples exposed to UV radiation in artificial chambers. The degradation due to weathering was successfully modeled using NIR hyperspectral imaging, which shows that this non-destructive technique is highly performant for these purposes. Regression models with good predictive abilities were obtained using Tikhonov regression with the total amount of UV exposure as response variable. Thermogravimetric analysis of a selection of the weathered samples demonstrated a clear trend in the mass loss curves with the amount of UV radiation, indicating that the lignin degradation of weathered samples could also be modeled from NIR spectroscopy. However, further thermogravimetric studies combined with density measurements of the samples needs to be carried out to confirm this.

The result from the study is a first step towards a weather dose response model determined by temperature and moisture content on the wooden surface in addition to the solar radiation. Such a model is expected to be essential for predicting the future performance of wooden façades' elements.

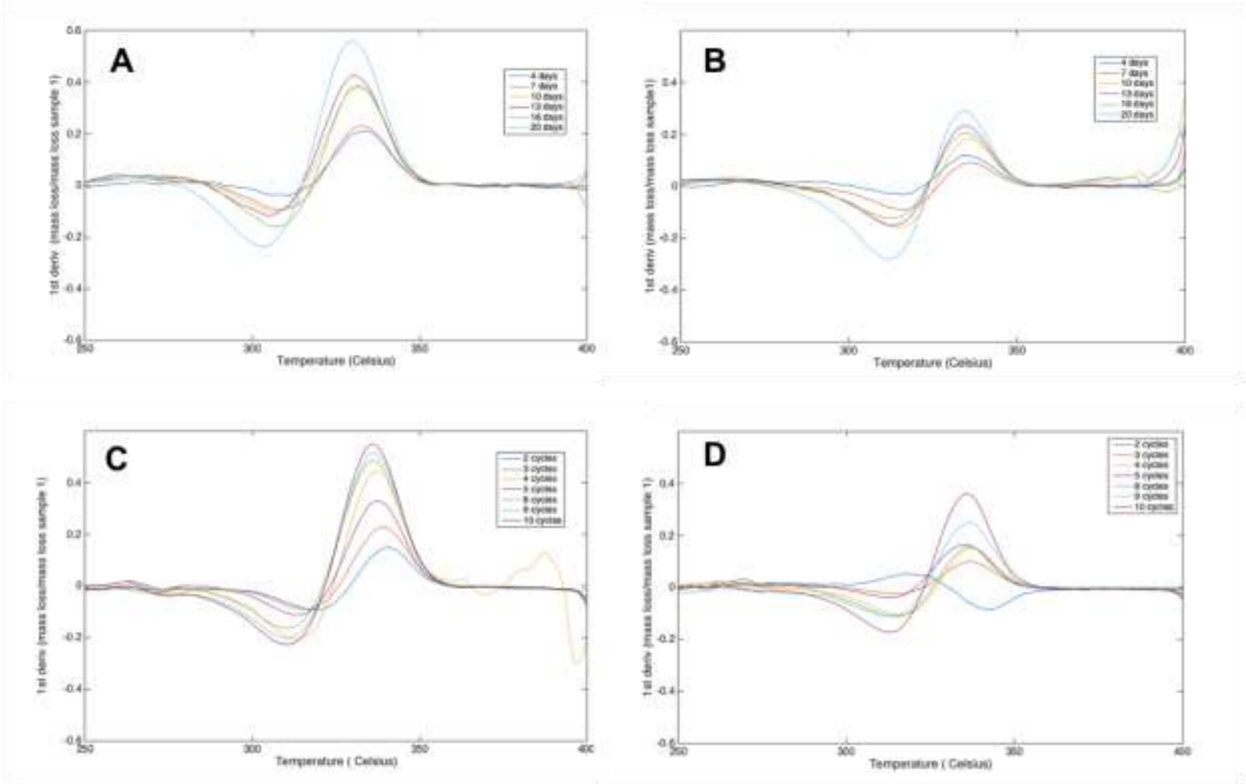


Figure 9: Mass loss relative to the first sample, corresponding to 1 day of outdoor exposure or 1 cycle in UV chamber. The plots are 1st derivatives of the thermogravimetric curves. A: earlywood exposed outdoor, B: latewood exposed outdoor, C: earlywood from UV chamber, D: latewood from UV chamber.

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