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Investigations into the photodegradation of wood using microtensile testing

Part 2: An investigation of the changes in tensile strength of different softwood species during natural weathering

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This paper describes changes in microtensile strength of six softwood species during natural weathering. All species showed a rapid reduction in both zero span and 10 mm span strength during three months weathering and the method proved capable of distinguishing differences in degradation rate and in the characteristic shape of the strength loss curve for the different species. The results indicated that lignin degradation is initially more rapid than cellulose. Although the principal trend was one of strength loss during weathering, some species showed a small strength increase after short exposure times.The strength changes are consistent with three radiation-induced processes, namely a mechanism whereby strength increases and two degradation processes occuring at different rates. A 'Zero Intercept' parameter was derived to characterise the strength loss curves.

Untersuchung des Photoabbaus von Holz mit Hilfe einer Mikro-Zugfestigkeitsprüfung. Teil 2: Änderung der Zugfestigkeit verschiedener Nadelhölzer während der natürlichen Bewitterung

Diese Arbeit beschreibt die Änderung der Zugfestigkeit von sechs Nadelholzarten während einer natürlichen Bewitterung über einen Zeitraum von drei Monaten. Bei allen Holzarten ergab sich eine rasche Abnahme der Zugfestigkeit sowohl bei Prüflänge Null als auch bei 10 mm Prüflänge. Die Methode erwies sich als geeignet, Unterschiede in der Abbaurate und im Verlauf des Festigkeitsverlustes für die verschiedenen Holzarten zu erfassen. Die Ergebnisse deuten daraufhin, daß Lignin zunächst rascher abgebaut wird als Cellulose. Entgegen dem allgemeinen Trend eines kontinuierlichen Festigkeitsbverlustes während der Bewitterung zeigten einige Holzarten einen vorübergehenden Anstieg nach kurzer Bewitterungsdauer. Die Festigkeitsänderungen stehen in Zusammenhang mit drei durch Lichtstrahlung induzierten Prozessen, von denen einer zur Erhöhung der Festigkeit führt. Zwei andere beruhen auf Abbauvorgängen mit unterschiedlicher Geschwindigkeit. Aus den Meßdaten wurde ein spezieller Parameter (Zero Intercept) zur Charakterisierung der Abbaukurven entwickelt.

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1 Introduction

Part 1 in this series of papers (Derbyshire et al. 1995) demonstrated that the measurement of the loss of microtensile strength of thin wood strips exposed to solar radiation is a consistent and reliable means of determining photodegradation rates for wood. With careful selection and preparation of material it was shown that levels of variability could be kept low and reproducible results obtained.

The application of the Pulmac short span paper tester to the tensile testing of the thin wood strips was shown to be a considerable advance in the technique. Tensile tests were carried out on the wood strips at zero span and at 10 mm span thus increasing the amount of information available from the test as well as providing savings in operating time and material requirements. It was established that the losses in tensile strength during exposure recorded in this way were fully compatible with earlier measurements obtained using conventional tensile testing techniques (Derbyshire, Miller 1981); the results reported by Derbyshire et al. (1995) confirmed the earlier finding by Derbyshire and Miller (1981) that the strength loss was an exponentially decreasing function of radiation dose, characterised by a degradation rate.

This paper presents a comparison of the strength losses of different softwood species during natural weathering. The results showed that the simple exponential relation described above did not always accurately describe the losses in tensile strength and an alternative theoretical approach is proposed.

2 Experimental

2.1 Timber

The following six softwood species were used in the investigation:

Scots pine sapwood (*Pinus sylvestris*)
Norway spruce (*Picea abies*)
Sitka spruce (*Picea sitchensis*)
Douglas fir (*Pseudotsuga menziesii*)
Hemlock (*Tsuga heterophylla*)
Sequoia (*Sequoia sempervirens*)

The physical characteristics and tensile strength of the unweathered timbers, as determined after conditioning at 20 °C, 65% relative humidity, are detailed in Table 1.

2.2 Preparation of thin strips

The procedure for preparation of the microtomed strips was fully reported in Part 1; a brief description is given here. Blocks of dimensions 100 mm × 10 mm × 20 mm in the longitudinal, radial and tangential directions respectively were vacuum impregnated with distilled water at ambient temperature

Table 1. Characteristics of softwood species tested

Species	Density kg/m³ (at 20 °C, 65% rh)	No. of latewood bands per 10 mm width	Tensile strength MPa	
			Zero span	10 mm span
Scots pine sapwood	528	4	61.6	51.4
Norway spruce	431	7	85.8	69.5
Sitka spruce	401	3	93.0	73.1
Douglas fir	465	approx. 20	68.2	21.1
Hemlock	510	approx. 15	78.8	58.0
Sequoia	379	approx. 20	41.5	23.3

(approximately 20 °C) until fully saturated. Thin strips were then microtomed from the radial face of the block using a conventional sliding microtome. For Scots pine sapwood, Norway spruce and Sitka spruce strips of thickness 80 μm were cut; for Douglas fir, Hemlock and Sequoia the strip thickness was increased to 95 μm as this produced strips of more uniform thickness. After drying, the strips were checked for uniformity in thickness using a Mercer electronic thickness gauge. Tensile tests were carried out on batches of 5 strips from each species to determine the initial values of zero span and 10 mm span tensile strength.

2.3
Weathering

A total of 6 batches of 5 strips of each species was exposed to natural weathering from 3 June to 7 September 1987 at the exposure site of the former Princes Risborough Laboratory of the BRE. The site is inland, semi-rural, situated at 52° latitude and 100 m above sea level. The strips were held in place in Perspex frames, which were mounted in shallow, matt black metal trays fitted with thin glass lids. Direct access by rainfall was thus prevented and some limited air circulation was provided by ventilation slots in the base of the cells. The ultraviolet and visible radiation transmission characteristics of the glass lids indicated a level of 90% transmission in the wavelength range 315–800 nm. The cells were mounted on racks at 45° to the vertical facing South.

2.4
Measurement of radiation dose

Full details of the method used to record levels of ultraviolet (UV) radiation during exposure are to be found in Part 1. The change in optical density of a poly(phenylene oxide) film sensor (Davis et al. 1976) was used as a measure of UV dose, radiation being recorded in Black Lamp Equivalent Time Units (BLETs). As described in Part 1, this could be related to the active solar energy received, the conversion factor being 64.3 Wh/m² per BLET.

2.5
Microtensile strength measurements

Tensile strength measurements were carried out on dry samples to determine the ultimate breaking load at zero span and at 10 mm span. Tests were carried out in standard laboratory conditions of 20 ± 1 °C, 60 ± 5% relative humidity. The batch of strips was cut so that each of the five strips provided 2 samples for the zero span test and 2 for the 10 mm span test. By this means the value of tensile strength was the mean of 10 replicate tests.

A full discussion of the operation of the Pulmac tensile testing machines was given in Part 1. A constant loading rate of 70 kPa/s was used and clamping pressures lay in the range 400–700 kPa. For the reasons given in Part 1 tensile strength values were expressed in terms of stress in MPa (1 MPa = 1 N/mm²) only for unweathered material. In monitoring photodegradation, tensile strength changes were expressed as a percentage of the initial breaking load.

3
Results

Graphs showing the loss in zero span and 10 mm span strength for all six species are presented in Figs. 1a–1f. The total radiation dose received in the full thirteen week exposure period amounted to 86 kWh/m². During that time six withdrawals were made. The graphs in Fig. 1 show that, with the exception of Douglas fir, the initial decrease in tensile strength was more rapid in the 10 mm span tests than in zero span tests. Since the zero span tensile test is essentially a measure of fibre strength and the 10 mm span test is determined additionally by network effects (Derbyshire et al. 1995), this observation is indicative of a greater sensitivity of the lignin-rich intercellular material to photodegradation. However after a radiation dose of approximately 50–60 kWh/m² similar values of strength retention were recorded in both zero and 10 mm span tests indicating substantial degradation of both lignin and cellulose.

Table 2 presents the degradation rate constant associated with each of the strength loss curves. Rates were calculated from the exponential relation between strength and dose (Derbyshire, Miller 1981):

$$S = S_0 \exp(-kD) \tag{1}$$

where S is the tensile strength after exposure to a radiation dose D and S_0 the initial tensile strength of the strips prior to weathering. The constant k in the exponent is the degradation rate and has units of inverse dose, in this case $(\text{kWh/m}^2)^{-1}$. The values of degradation rate in Table 2 were calculated from a linear regression analysis of $\text{Ln}(S/S_0)$ on D and the regression coefficients indicate the goodness of fit of the data to the exponential equation.

It will be seen from Table 2 that the differences in degradation rates for the different softwood species were not great and in general the zero span and 10 mm span rates were similar for each species. Only Douglas fir showed a large difference between the two degradation rates, with the value of 0.0071 $(\text{kWh/m}^2)^{-1}$ for the 10 mm span test being unusually low. The most rapid degradation occurred in Sitka spruce with rates of 0.0224 and 0.0258 $(\text{kWh/m}^2)^{-1}$ being recorded for the zero and 10 mm span tests respectively. Sequoia was the species which showed the lowest rates of degradation with the zero and 10 mm span tests yielding values of 0.0132 and 0.0131 $(\text{kWh/m}^2)^{-1}$ respectively.

With the exception of Douglas fir, the regression coefficients for the 10 mm span tests were better than those of the zero span tests. However the regression coefficients for all tests, except the 10 mm span test in Douglas fir, were in excess of 0.90 suggesting good fit of the experimental data to the exponential strength loss expression (Eq. 1). Despite these high values of

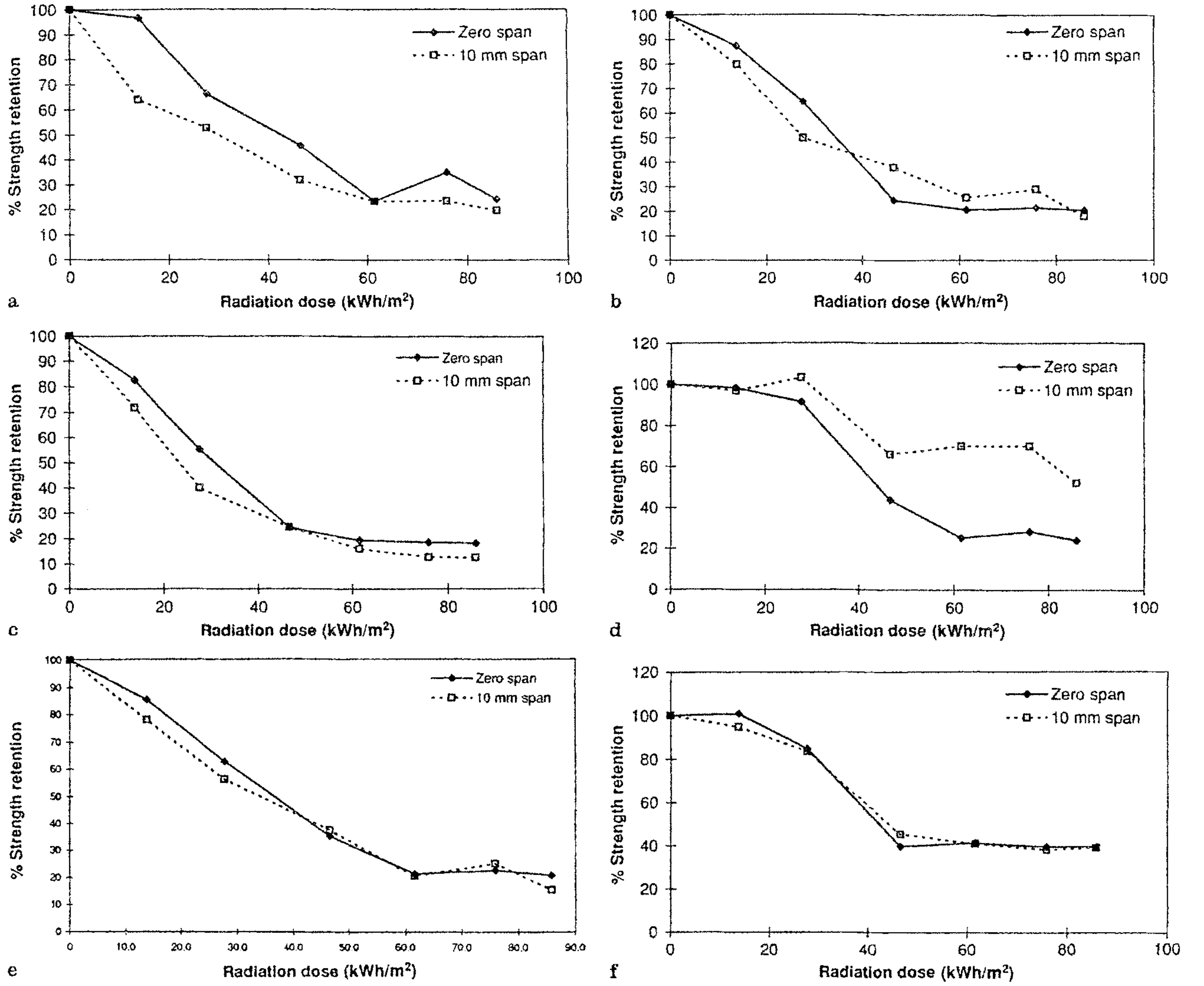

Fig. 1a–f. Changes in tensile strength during natural weathering. **a** Scots pine sapwood; **b** Norway spruce; **c** Sitka spruce; **d** Douglas fir; **e** Hemlock; **f** Sequoia

Bild 1a–f. Verlauf der Zugfestigkeit während künstlicher Bewitterung. **a** Kiefernsplintholz; **b** Fichte; **c** Sitkafichte; **d** Douglasie; **e** Hemlock; **f** Sequoie

Table 2. Degradation rate constants for different softwood species

Species	Span (mm)	Degradation rate $(kWh/m^2)^{-1}$	Regression coefficient
Scots pine sapwood	0	0.0177	0.939
	10	0.0186	0.979
Norway spruce	0	0.0215	0.943
	10	0.0190	0.976
Sitka spruce	0	0.0224	0.961
	10	0.0258	0.985
Douglas fir	0	0.0196	0.952
	10	0.0071	0.894
Hemlock	0	0.0205	0.972
	10	0.0213	0.977
Sequoia	0	0.0132	0.915
	10	0.0131	0.949

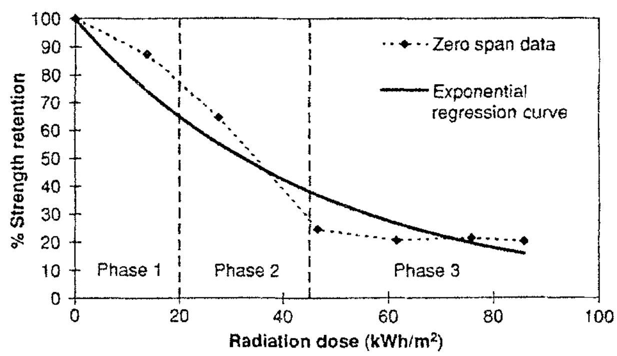

Fig. 2. Comparison of exponential decay curve with experimental data for Norway spruce

Bild 2. Exponentieller Abbau (Regressionskurve) und Meßdaten and Fichtenholz

the regression coefficients it was felt that the calculated exponential decay curve did not reflect the shape of many of the strength loss curves shown in Figs. 1a–f. This was especially the case for the zero span tests which frequently showed a shoulder, or in some cases a small increase at the start of the degradation curve. Figure 2 shows the calculated exponential regression line through the zero span data for Norway spruce. The data depart from the calculated curve in three ways. Firstly the initial rate of decrease is slower than exponential (marked as phase 1 in Fig. 2). Secondly the subsequent rate of decrease is faster than the calculated exponential (phase 2). Thirdly in the final stages of degradation the observed rate of strength decrease is very slow indeed (phase 3). This pattern of behaviour was observed consistently in the zero span results and in some cases in the 10 mm span results as well.

4
A mathematical model of the strength changes

The observed strength changes of the zero span tests suggest three distinct processes combining to produce the strength loss curve. The observation of the shoulder or small increase in strength in the first phase of degradation indicates that in addition to the degradation process which results in a reduction of tensile strength, there is a radiation-induced process which results in an increase in strength. This could be, for example, a cross-linking mechanism. It is suggested that this process could be expressed mathematically in the form:

$$S = S_0(1 + C(1 - \exp(-bD))) \quad (2)$$

where S is the tensile strength at radiation dose D, S_0 is the initial value of tensile strength prior to radiation. The constant b in the exponent would determine the rate of strength increase and the factor C, also a constant, would define the limiting value of strength. For example a value of 0.25 for C would correspond to a limiting strength value 25% higher than the initial value.

The different rates of decrease which seem to be associated with phases 2 and 3 of the strength loss curve suggest the presence of two components within the wood which degrade at different rates. These two components could be interpreted, for example, as being lignin and cellulose since it is known that lignin is more susceptible to photodegradation than cellulose (Hon, Shiraishi 1991; Evans et al. 1992). The results reported here of the more rapid loss of strength in the early stages of degradation of the 10 mm span tests compared with the zero span tests are further evidence of the more rapid degradation of lignin. Alternatively, the two components could be associated with the earlywood and latewood regions of growth; again it has been shown that earlywood degrades more rapidly than latewood (Derbyshire, Miller 1981).

This two-component interpretation of the degradation process would be expressed mathematically by an adaptation of Eq. (1):

$$S = S_0((1 - f)\exp(-k_1D) + f\exp(-k_2D)) \quad (3)$$

Here k_1 represents the rate of degradation of the photochemically more susceptible component and k_2 that of the photochemically more resistant component. The constant f is a weighting factor, expressing the quantity of the photochemically resistant material as a fraction of the total wood substance. It could relate for example to the fraction of latewood present or the cellulose content of the sample.

Combining the mathematical expressions for the strength increase (Eq. 2) with that of the strength decrease (Eq. 3), the full expression for the strength change would become:

$$S = S_0\{1 + C(1 - \exp(-bD))\} \times \{(1 - f)\exp(-k_1D) + f\exp(-k_2D)\} \quad (4)$$

5
Comparison of the model with experimental results

5.1
Method of analysis

The proposed Eq. (4) was too complex for regression analysis to be applied to the data analysis and the following alternative approach was accordingly adopted. A trial set of the parameters C, b, k_1, k_2 and f was chosen and a graph plotted showing the experimental data together with the curve generated by Eq. (4). The parameters were then adjusted to produce the curve which best fitted the experimental data. The curve which best fitted the data was judged by eye and the goodness of fit was measured using a value termed the residual coefficient. This was calculated from RSS, the sum of the squares of the residuals (i.e. the difference between the experimental value and the calculated value). The residual coefficient was then defined to be:

$$(1 - RSS)^{0.5}$$

Thus, like the regression coefficient, a residual coefficient close to 1 indicates a good fit.

5.2
Results of analysis

The results of the analysis of the zero and 10 mm span strength loss curves for the six different species are presented in Table 3.

Table 3. Parameters for strength change during weathering

Species	Span (mm)	b	k_1	k_2	residual coefficient [Eq. (4)]	exponential residual coeff. [Eq. (1)]
Scots pine	0	0.14	0.037	0.014	0.982	0.965
sapwood	10	0.01	0.046	0.014	0.994	0.978
Norway spruce	0	0.15	0.045	0.019	0.985	0.967
	10	0.08	0.060	0.015	0.991	0.985
Sitka spruce	0	0.09	0.055	0.020	0.990	0.979
	10	0.04	0.060	0.022	0.996	0.987
Douglas fir	0	0.08	0.031	0.015	0.962	0.931
	10	0.04	0.071	0.006	0.909	0.887
Hemlock	0	0.055	0.035	0.020	0.992	0.986
	10	0.03	0.035	0.020	0.994	0.993
Sequoia	0	0.08	0.025	0.013	0.953	0.933
	10	0.08	0.027	0.012	0.971	0.958

Note:
b: exponential constant associated with radiation-induced strength increase
k_1: degradation rate associated with photochemically susceptible material
k_2: degradation rate associated with photochemically resistant material

Calculations assume a limiting value of 40% strength rise and a value of 0.45 for the fraction of photochemically resistant material for all species

For Sequoia and Douglas fir which showed a pronounced shoulder at the start of the degradation curve, it appeared that the value chosen for C needed to be as high as 0.4 in order to achieve the correct fit. For other species where the shoulder was less pronounced the value of C was not critical; however for consistency, a value of 0.4 for C was applied to these species also. A value of 0.45 was chosen for f, the proportion of photochemically resistant material as this was a typical value for cellulose content and latewood fraction; the value chosen for f did not appear to be critical. The values determined for b, k_1 and k_2 differed for each set of data and are shown in the table.

It was clear that the value of b, the rate associated with the radiation-induced strength increase, affected the shape of the curve in phase 1 of the degradation. Those results showing a high value for b relative to the degradation rates k_1 and k_2 were associated with the shoulder in the strength loss curve. The inclusion of the second degradation rate k_2 accounted for the slower rates of degradation in phase 3 of the strength loss curve observed in some tests. The expression given in Eq. (4) gave a better representation of the zero span strength change curves than did the simple exponential expression (Eq. 1). This is shown in Fig. 3a where the calculated curve based on Eq. (4) and the exponential curve based on Eq. (1) are compared with the results for the zero span test on Scots pine sapwood. It was also noteworthy to observe that the full expression of Eq. (4) also gave a better fit for the 10 mm span data, even where this showed no sign of a shoulder at the start of weathering. Figure 3b shows the case of the 10 mm span test on Scots pine sapwood. Figure 3b also illustrates a further significant advantage of using Eq. (4), namely that the initial value of tensile strength calculated for zero dose is always 100%; this is not necessarily the case when Eq. (1) is used to represent the data. The values for the residual coefficients for analysis by Eq. (4) and Eq. (1) are included in Table 3 and the better fit achieved by Eq. (4) is evident in the results.

5.3
Characterisation of the strength loss data

Although Eq. (4) appears to be a good representation of the strength changes during weathering, the expression is complex and requires several parameters to define it. Furthermore it is not possible to define the parameters uniquely in any one case, since similar curves could be generated from different sets of parameters.

A single quantity is required to characterise the strength loss curve and to allow comparison of different test results. To meet this need the concept of the Zero Intercept (ZI) was introduced. This was defined to be the point at which the tangent to the calculated curve at 50% strength retention intercepted the radiation dose axis, as shown in Fig. 4 for the case of Scots pine sapwood zero span test. This quantity is considered to be a good representation of the curve in that it is directly related to the degradation rate in phase 2 of the curve and also reflects the shape of the curve in phase 1; for a given rate of degradation in phase 2, the presence of a shoulder in phase 1 of the curve would increase the ZI value.

The calculated values of ZI for all the species are shown in Table 4. As was the case for the exponential degradation rates the differences between species were not great and the values for zero and 10 mm span tests of each species were broadly similar. It appeared that low values of ZI were associated with the higher values of degradation rate and conversely high values of ZI with low degradation rates. In Fig. 5 the reciprocal of the ZI is plotted against the exponential degradation rate calculated using Eq. (1) and there appeared to be a linear relation

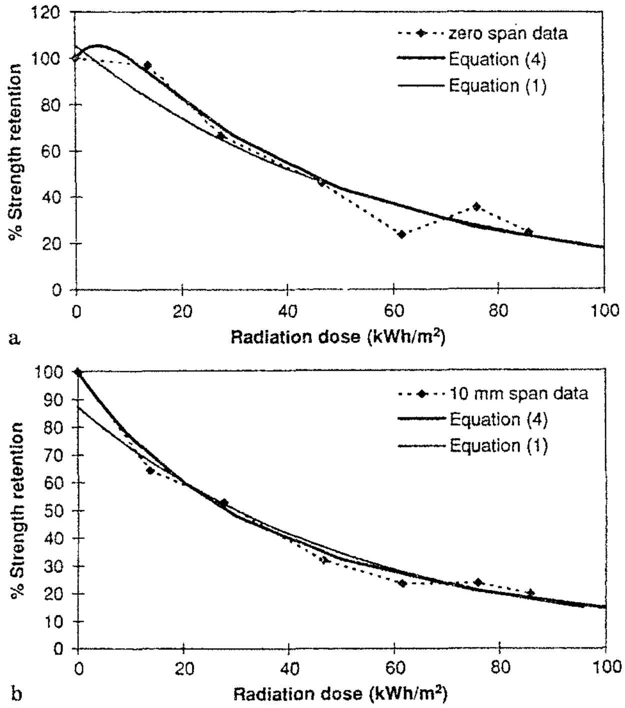

Fig. 3a, b. Comparison of mathematical expressions for strength change with experimental data for pine sapwood. a zero span data; b 10 mm span data
Bild 3a, b. Meßdaten und Regressionskurve der Festigkeitsänderung an Kiefernsplintholz. a Messung bei Prüflänge Null; b Messung bei Prüflänge 10 mm

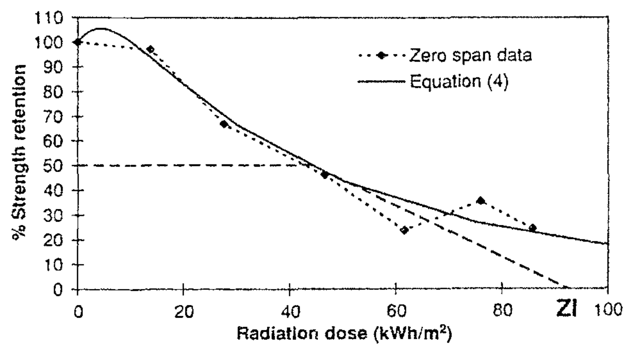

Fig. 4. Derivation of the Zero Intercept (ZI, Eq. 4), shown for pine sapwood
Bild 4. Verlauf des Parameters ZI (Zero Intercept, Gl. 4) am Beispiel von Kiefernsplintholz

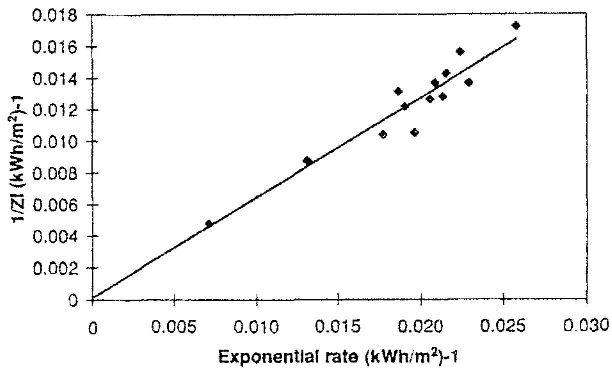

Fig. 5. Correlation of 1/ZI with degradation rate
Bild 5. Zusammenhang zwischen dem Parameter 1/ZI und der Abbaurate

Table 4. Calculated values of zero intercept for different softwood species

Species	ZI (kWh/m^2)	
	zero span	10 mm span
Scots pine sapwood	93	76
Norway spruce	71	76
Sitka spruce	64	58
Douglas fir	95	209
Hemlock	79	78
Sequoia	115	114

between them. Regression analysis confirmed this to be true with a high degree of correlation (coefficient 0.957).

6
Discussion

The proposal that the strength changes induced by exposure to natural weathering may be described by a combination of three exponential processes has led to the derivation of a new expression relating tensile strength to radiation dose, Eq. (4). This expression has been shown to agree well with experimental data, giving better residual coefficients than the original exponential decay expression, Eq. (1); the validity of the approach is thus supported. The indications that the radiation-induced strength changes may initially consist of a combination of strength increasing and strength decreasing effects are surprising. However Williams et al. (1990), in a study of the adhesion of paint to weathered wood surfaces, have reported that although paint adhesion was generally reduced by pre-weathering of the substrate, for some species short periods of pre-weathering actually increased adhesion. Kalnins and Knaebe (1992) studied the wettability of weathered timber by measurements of contact angle. They reported that for southern pine there was a general decrease in contact angle with weathering, although for short periods of exposure the contact angle increased. Finally a similar effect was observed by Horn et al. (1992) during FT-IR studies in weathered wood. These authors reported an increase in the intensity of the carbonyl band at $1750\,cm^{-1}$ for short periods of weathering followed by a decrease for longer exposure periods. They suggested that this change in intensity is not caused by a single process but that there may be competing mechanisms involved. The similarity of all these results with the initial increase in strength reported here is very striking and supports the suggestion that short periods of exposure to radiation could actually increase surface strength.

In many cases, although Eq. (4) gives a better fit to the data, the exponential decay curve given by Eq. (1) is a reasonably good fit. In these cases, the fact that the correlation between the reciprocal of the ZI value and the exponential degradation rate was so good, validates the use of ZI value as a single parameter to characterise the strength loss curve. This method of analysis could therefore be applied with confidence in cases where the simple exponential decay curve was clearly a poor representation of the strength changes.

The relatively small differences between the degradation rates and ZI values for the different species suggest that degradation processes are not strongly influenced by the differences in anatomical structure nor the nature and content of extractives. It should be noted that the rate of photodegradation indicates the rate at which the surface of wood deteriorates at relatively low moisture content during exposure to UV and visible light. It is not necessarily an indication of the likely durability of the timber which is determined by other factors such as the presence of fungi-toxic materials in the timber and permeability to water.

7
Conclusions

Changes in tensile strength during weathering have been measured for six different softwood species. With the exception of Douglas fir, the initial strength losses were greater in the 10 mm span tests than in the zero span test, indicating the degradation of lignin is initially more rapid than cellulose.

The strength changes during weathering were interpreted in terms of three radiation-induced processes. The mathematical expression derived from this approach fitted the experimental results well and the strength changes were characterised by the Zero Intercept.

The ZI values correlated well with rates of photodegradation calculated using the previously derived exponential decay equation. This suggests that the new expression proposed for describing the strength changes is a reliable means of assessing rates of photodegradation and would be a useful analysis tool where the exponential decay expression is inappropriate.

Although the differences in rates of photodegradation and ZI values for the different species do not seem great, they indicate that the reactions of the various species to the effect of light are different in magnitude and in character and that the thin strip test is capable of distinguishing them. Sitka spruce showed the most rapid rates of photodegradation (ZI values of 64 and $58\,kWh/m^2$ in the zero and 10 mm span tests) and Sequoia the slowest rates of degradation (ZI values of 115 and $114\,kWh/m^2$ in the zero and 10 mm span tests). The result for the 10 mm span test on Douglas fir yielded an unusually high ZI value of $209\,kWh/m^2$.

8
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