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# Microtensile Testing of Wood – Influence of Material Properties, Exposure and Testing Conditions on Analysis of Photodegradation

## Ispitivanje mikrovlačne čvrstoće drva – utjecaj svojstava materijala, izlaganja i uvjeta ispitivanja na fotodegradaciju

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**ABSTRACT** • This paper presents the effects of properties of tested material and exposure conditions on the final result of testing. These include density, uniformity of ring width, number of rings and latewood portions, as well as light source, presence of water and duration of exposure. Influences of these parameters in testing of several softwood species after exposure to different natural and artificial photodegradation regimes were monitored by means of changes in microtensile properties. The main findings indicate that comparisons between various species should be made taking into account average material properties, predominantly latewood portion. The fact that strength loss changes follow the same pattern during different exposure conditions indicates that there is no difference in the nature of degradation process in various weathering machines. This forms a basis for the sound comparison between the artificial and natural weathering regimes.

**Keywords:** softwoods, microtensile testing, thin strips, density, photodegradation

**SAŽETAK** • U radu se opisuje istraživanje utjecaja svojstava ispitivanog materijala i uvjeta izlaganja drva na konačni rezultat ispitivanja fotodegradacije njegova površinskog sloja. Ispitivana svojstva materijala obuhvaćaju gustoću drva, ujednačenost širine goda, broj godina i udio kasnog drva, utjecaj izvora zračenja, postojanje vode i duljinu izlaganja vremenskim utjecajima. Mjerenje mikrovlačne čvrstoće u smjeru drvnih vlakana ispitano je na više vrsta drva nakon prirodnoga i ubrzanih umjetnih izlaganja vremenskim utjecajima. Rezultati pokazuju kako je za valjanu usporedbu različitih vrsta drva potrebno odabrati materijal prosječne gustoće, posebno pazeći na udio kasnog drva. Činjenica da je gubitak čvrstoće tijekom različitih vrsta izlaganja podjednak upućuje na zaključak kako nema bitne razlike u prirodi procesa površinske degradacije ovisno o vrsti izvora zračenja. To je osnova za valjanu usporedbu rezultata dobivenih izlaganjem u različitim prirodnim i umjetnim uvjetima.

**Ključne riječi:** četinjače, mikrovlačno ispitivanje, tanki listići, gustoća, fotodegradacija

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## 1 INTRODUCTION

### 1. UVOD

Properties of tested material as well as weathering conditions strongly affect the reliability of microtensile testing and its final result. Derbyshire *et al.* (1995, 1996) demonstrated that the measurement of loss of microtensile strength of thin wood strips exposed to solar radiation offers a consistent, reliable and precise means of determining photodegradation rates for wood. It was shown that artificial weathering regimes could provide good simulation of the effects of natural weathering, and mathematical analysis procedures were developed to characterize the strength changes observed during weathering. In the third paper in a series, Derbyshire *et al.* (1997) further showed that photodegradation rates of a number of different softwood species during artificial weathering were temperature dependent, increasing with rising temperature. In an additional paper in that series (Part 4: Turkulin and Sell, 2002), the strength changes were shown to be consistent with fractographic evidence of the structural changes in wood, namely with cell delamination, development of brittleness and loss in cell wall integrity. In the last paper in that series (Turkulin *et al.*, 2004), the thin strip technique was used to investigate the effect of moisture on photodegradation rates. Moisture is a significant factor in accelerating photodegradation of wood by means of increasing the rates of strength loss.

Another interesting finding demonstrated that the exposure of the strips at high levels of relative humidity could result in an initial increase in tensile strength, evident in the form of a shoulder or small initial positive peak in the strength loss curves. This strength increase appeared to be associated with cellulose changes, since it is regularly recorded in testing over zero initial span of clamps of the testing instrument, and is believed that it reflects some form of cellulose crosslinking at shorter exposure times, followed by the general strength loss that develops at prolonged exposure to elements (Derbyshire *et al.*, 1996; Turkulin and Sell, 2002). Differences in photoresistance between the different wood species, and between heartwood and sapwood, were most notably visible under dry exposure conditions. These differences progressively diminished as the pronounced effect of water was introduced in subsequent weathering regimes.

The thin strip method was also successfully applied in studies of weathering of acetylated and thermally modified wood surfaces (Evans *et al.*, 2000; Altgen and Militz, 2016), determination of the depth profile of weathering effects on unprotected and protected softwoods (Jirouš-Rajković *et al.*, 2004; Kataoka *et al.*, 2005), effects of chemical modifications on its mechanical properties (Bischof Vukušić *et al.*, 2006; Xie *et al.*, 2007), the degradation effects of wood destroying fungi (Lehringer *et al.*, 2011), effects of seawater wetting on the weathering of wood (Kluppel and Mai, 2017) as well as spectral sensitivity and depth profiling during photodegradation of fir wood surfaces (Živković *et al.*, 2014 and 2016).

This paper presents the effects of selected properties of tested material on the final result of testing. Variables included the specific anatomical and physical properties of several wood species, namely the density, ring width, number of rings and latewood portions. Other parameters that varied in a series of experiments involved weathering conditions - particularly the light source, presence of water and duration of exposure. Results presented here demonstrate specific aspects of the thin strip method as a useful tool in the analysis of photodegradation of wood surfaces and its applicability to other studies on wood surfaces.

## 2 MATERIALS AND METHODS

### 2. MATERIJALI I METODE

#### 2.1 Material characteristics

##### 2.1. Svojstva materijala

Radial strips of nominal thickness of 80  $\mu\text{m}$  were microtomed from Scots pine sapwood – SPS (*Pinus sylvestris* L.); European spruce (*Picea abies* Karst.), in further text Norway spruce – NS and Croatian spruce – CS, according to the place of origin; and European Silver Fir (*Abies alba* Mill.) marked as Croatian fir – CF that was selected as fast grown – FG and slow grown – SG. An overview of the basic physical and mechanical properties of the tested material is given in Table 1. The testing material has been deliberately selected from the commercial stock so that the recorded changes could be associated with the standard processed wood products. Optimal thickness for the investigation of the wood surface photodegradation is the depth to which light and elements will cause the greatest effect, which was shown to be ca 70 – 80  $\mu\text{m}$  for temperate softwoods. This thickness is also practical from the experimental point of view - resulting in no slipping of the samples and achieving the optimal range of ultimate load values when tested with short span tensile tester. Detailed elaboration on the issue of specimen geometry is presented by Živković and Turkulin (2014).

The basic physical and mechanical properties of tested species are given in Table 1. The procedures for the preparation and testing of the radial and tangential thin strips were fully reported and analyzed earlier (Derbyshire *et al.*, 1995, 1996; Živković and Turkulin, 2014) and only a brief description is given here.

The strips were stored in the dark prior to and after exposure, and handled in controlled and constant atmospheric conditions of  $20 \pm 2$  °C and  $60 \pm 5$  % relative humidity.

Codes in the first column of Table 1 are used throughout the text as acronyms for wood species. The conditions and codes for weathering regimes are given in Table 2.

#### 2.2 Exposure conditions and specimen handling

##### 2.2. Uvjeti izlaganja i rukovanje uzorcima

Strips were mounted on frames made of 1.2 mm thick aluminum sheets with two rectangular openings. A batch of five strips was attached over each of these open-

**Table 1** Basic physical and mechanical properties of the testing material  
**Tablica 1.** Osnovna fizikalna i mehanička svojstva ispitivanog materijala

| Species code<br><i>Oznaka vrste drva</i> | Average density at 12 % m.c.<br><i>Prosječna gustoća pri 12 % s.v.</i> | Strip thickness range<br><i>Raspon debljine listića</i> | Growth rings<br><i>Godovi</i> |                                      | Latewood<br><i>Kasno drvo</i> |                           | Initial tensile strength at chosen testing span*<br><i>Početna čvrstoća na vlak na izabranom rasponu čeljusti*</i> |                   |                             |        |
|--|--|---|-------------------------------|--------------------------------------|-------------------------------|---------------------------|--|-------------------|-----------------------------|--------|
|  |  |   | Width<br><i>Širina</i>        | Nr per 10 mm<br><i>Broj na 10 mm</i> | Width<br><i>Širina</i>        | Proportion<br><i>Udio</i> | 0 mm Span<br><i>Razmak</i>   | C.V.**            | 10 mm Span<br><i>Razmak</i> | C.V.** |
|  |  | Min - Avg - Max   | mm                            | mm                                   | %                             | N/mm <sup>2</sup>         | %  | N/mm <sup>2</sup> | %                           |        |
| SPS1                                     | 440±10   | 70 - 78.6 - 90  | 2.56                          | 4                                    | 0.95                          | 38                        | 89   | 7.2               | 50                          | 9.1    |
| SPS2                                     | 540±10   | 67 - 68.7 - 73  | 2.45                          | 4                                    | 0.88                          | 36                        | 90   | 8.1               | 66                          | 9.6    |
| NS                                       | 369±10   | 70 - 81.5 - 90  | 3.33                          | 3 - 4                                | 1.58                          | 48                        | 82   | 11.3              | 58                          | 12.5   |
| CS                                       | 483±10   | 65 - 72.3 - 75  | 2.60                          | 4                                    | 1.01                          | 38                        | 85   | 8.5               | 67                          | 11.3   |
| CFSG                                     | 510±20   | 70 - 79.4 - 90  | 1.36                          | 6 - 7                                | 0.48                          | 35                        | 138  | 5.7               | 102                         | 11.7   |
| CFFG                                     | 545±20   | 70 - 81.5 - 90  | 2.45                          | 3 - 4                                | 1.12                          | 46                        | 125  | 9.1               | 103                         | 14.16  |

\*initial strength was calculated for initial testing only on the basis of the geometrical cross-section of the strips. Further on, only the retained percentage of initial load to failure was used as indicator of strength changes that form the strength-loss curves. / *Početna čvrstoća izračunana je na početku istraživanja samo na temelju geometrijskoga poprečnog presjeka listića. Nadalje, samo je zadržani postotak početnog opterećenja do loma korišten kao pokazatelj promjena čvrstoće koji čine krivulje gubitka čvrstoće* \*\* C.V. is abbreviation for the Coefficient of variation. / *C.V. kratica je za koeficijent varijacije.*

ings using double-sided adhesive tape. Aluminum frames were generally backed with aluminum backing panels in close contact with the strips in order to keep the control of the chamber conditions, since the space behind the panels was ventilated by room air to activate condensation on the strips in cycle QUV 2. The strips exposed to high humidity (QUV3 cycle and natural exposure) were mounted on aluminum frames using 3 mm thick double-sided adhesive tape spacer to avoid any formation of liquid (droplets or accumulation at the bottoms) on the material. Metal backing plates were used consistently throughout the experiment to reflect the light to the back of the strips and seal the chamber and ensure that the desired conditions are met.

Specimens of wood species listed in Table 1 were either exposed to some of artificial weathering regimes (UV-fluorescent light, further denoted as “QUV”) or

boron-glass filtered xenon-arc light (in further text “XT”) or to natural weathering (NE). The overview of specific weathering conditions is given in Table 2. Batches of strips (usually 9 batches of five strips) were withdrawn at intervals adjusted to the expected development of degradation in particular exposure regime. Intervals were shorter in initial phases and gradually longer with development of exposure duration until the strips were degraded beyond the point of physical coherence. At the end of natural exposure and exposure to wet conditions, the earlywood bands of the strips were fully disintegrated. The effect of selected conditions on photo-degradation rates was readily monitored after each withdrawal.

Strips for natural weathering were backed with white filter paper and exposed horizontally on the exposure site at the Building Research Establishment (BRE,

**Table 2** Conditions and technical details used in artificial weathering regimes  
**Tablica 2.** Uvjeti i tehnički detalji ubrzanog izlaganja uzoraka vremenskim utjecajima

| Weathering code<br><i>Oznaka izlaganja</i> | Weathered wood species<br><i>Vrsta drva</i> | General conditions<br><i>Opći uvjeti</i> | Light source<br><i>Izvor svjetlosti</i> | Conditions in exposure regimes<br><i>Uvjeti tijekom izlaganja</i> |   |  | Sample mounting details<br><i>Pozicioniranje uzoraka</i> |                           |
|--|---|--|---|---|---|--|--|---------------------------|
|  |   |  |   | Chamber Temp.<br><i>Temperatura komore</i><br>°C                  | Relative Humidity in chamber<br><i>Relativna vlažnost zraka u komori</i><br>% | Black Panel Temp.<br><i>Temperature crne ploče</i><br>°C | Exposure setting<br><i>Vrsta izlaganja</i>               | Backing<br><i>Oslonac</i> |
| QUV-1                                      | CFSG, CFFG                                  | Constant dry                             | UVA 340                                 | 57 - 65   | 26 - 30   | 56 - 59  | Single-sided   | Al panel in contact       |
| QUV-2                                      | CS, NS, CFSG, CFFG, SPS                     | High humidity                            | UVA 340                                 | 57 - 59   | 80 - 100*   | 57 - 60  | Single-sided   | Al panel no contact       |
| QUV-3                                      | CS, SPS                                     | High humidity                            | UVA 340                                 | 57 - 59   | 80 - 100*   | 57 - 60  | On both sides  | No backing                |
| XT   | CFSG, CFFG, SPS                             | Moderate humidity                        | Xenon (4.5 kW)                          | 55 - 59   | > 90  | 78   | On both sides  | No backing                |

\*These conditions were achieved and continuously maintained during exposure. / *Ti su uvjeti postignuti i tijekom izlaganja održavani stalnima.*

Watford, England 52° N and 70 m above sea level) for a period of 3 months during summer (warm continental climate with incidental rain spells).

After withdrawal, the strips were allowed to recondition in room conditions on the frame, and stored in the dark. They were removed from the frame by rocking action of the oval scalpel immediately before tensile testing, and were cut in minimum 10 specimens for each testing span (0 mm and 10 mm).

## 2.3 Testing and presentation of results

### 2.3. Ispitivanje i prikaz rezultata

Tensile tests were carried out on dry samples, after conditioning in the dark at  $20 \pm 1^\circ\text{C}$  and  $60 \pm 5\%$  RH using a short span tensile tester. The exception to this procedure was with Croatian spruce, which was tested both dry and wet (Živković and Turkulin, 2014). Tests were carried out at 0 mm span and 10 mm span (details presented by Živković and Turkulin, 2014). Graphs showing the loss in zero and 10 mm span strength for all species presented in this paper are plotted according to the following expression:

$$S = S_0 \{1 + C(1 - \exp(-bD))\} \times \{(1 - f) \exp(-k_1 D) + f \exp(-k_2 D)\}$$

Where  $S$  is the tensile strength at radiation dose  $D$ ,  $S_0$  is the initial value of tensile strength prior to exposure. The constant  $b$  is the rate of strength increase and the constant  $C$  is the limiting value of strength,  $k_1$  is the rate of degradation of the photochemically more susceptible component and  $k_2$  that of the photochemically more resistant component. Constant  $f$  is a weighing factor presenting the quantity of the photochemically resistant material as a fraction of

the total wood substance. This equation generally enables the interpretation of three distinct, yet combined processes involved in photo-induced wood decomposition: there are two different rates of degradation (the first, more intensive, associated with delignification, and the later, slower rate associated with carbohydrate decomposition). Additionally, there may appear a short-lived initial antagonistic process of strength increase. A detailed formulation of this mathematical model is described by Derbyshire *et al.* (1996).

In all of the plots, the correlation factors between the curves plotted on the basis of measured and calculated values exceeded 0.96, and in vast majority of cases were higher than 0.98.

## 3 RESULTS

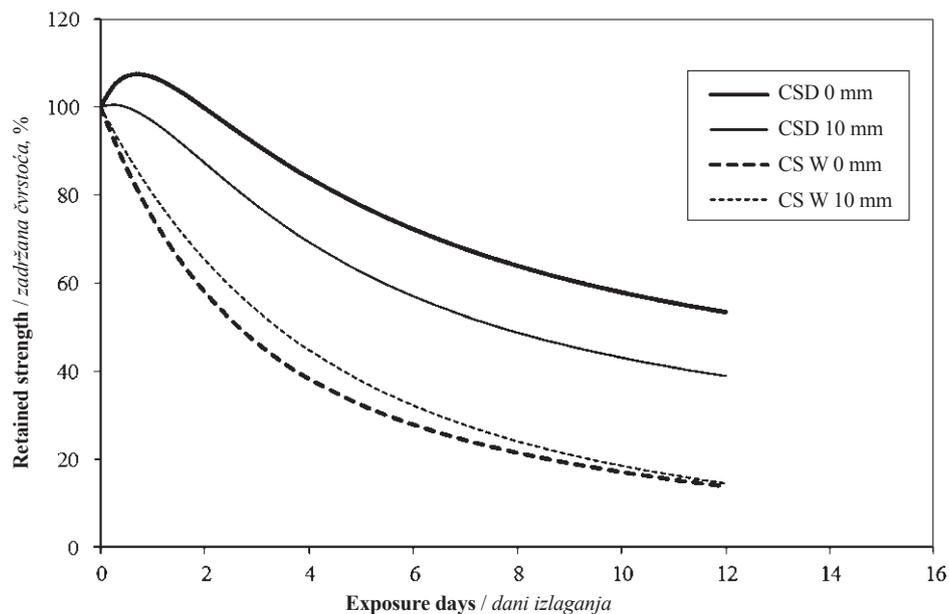
### 3. REZULTATI

#### 3.1 Dry / Wet testing

##### 3.1. Ispitivanje u suhim i mokrim uvjetima

Wet testing gives the same initial value of the zero span ultimate load, but 30 % smaller values in 10 mm span test. This indicates the effect of the penetration of water into the intercell regions and probably onto the fibre interfaces.

The character of tensile changes over the time of exposure and results in dry and wet conditions, i.e. after full aspiration of the specimens for at least half an hour prior to tensile testing, are presented in Figure 1. The dry testing shows very typical strength changes: in zero span testing, the strength initially rises forming a shoulder, then gradually drops. The finite span dry testing shows a slight delay of the strength loss rate in the second phase, then fairly stable strength loss.



**Figure 1** The effect of wet testing of Croatian spruce (CS) on the character of tensile changes during exposure to high humidity conditions (QUV-3). Specimens were tested in dry (D) conditions (at  $20 \pm 1^\circ\text{C}$  and  $60 \pm 5\%$  RH) and wet (W), i.e. after full aspiration of water

**Slika 1.** Učinak mokrog ispitivanja smrekovine (CS) na karakter vlažnih promjena tijekom izlaganja u uvjetima visoke vlažnosti (QUV-3). Probni su uzorci kidani u suhom stanju (pri  $20 \pm 1^\circ\text{C}$  i  $60 \pm 5\%$  r.v.z.) ili u mokrom stanju, tj. nakon potapanja u vodi

**Table 3** Dry to wet strength ratio of the Croatian spruce radial strips during weathering in QUV-3 cycle

**Tablica 3.** Odnos čvrstoće suhих i mokrih radijalnih listića jelovine tijekom izlaganja uvjetima u QUV-3

| Span / Raspon<br>mm | Dry / wet strength after exposure (days) / Čvrstoća suhих /čvrstoća mokrih listića nakon izlaganja (dani) |     |     |     |     |     |     |     |     |
|---------------------|---|-----|-----|-----|-----|-----|-----|-----|-----|
|                     | 0   | 0.5 | 1   | 1.5 | 2   | 2.5 | 3   | 4   | 7   |
| 0                   | ~1  | 1.3 | 1.5 | 1.5 | 1.6 | 1.7 | 2   | 2   | 3.2 |
| 10                  | 1.4   | 1.6 | 1.7 | 1.6 | 2   | 1.9 | 2.1 | 2.5 | 3.4 |

Wet testing strength loss curves show different characteristics of strength changes than the dry tensile changes:

- no initial effect on strength increase (shoulder) or delayed strength drop can be seen;
- strength loss rates are much higher than in dry testing in both span tests;
- dry to wet strength ratio progressively grows during exposure (Table 3) and the trend is similar in both span tests. As the wood deteriorates, its strength becomes more sensitive to the effect of water.

Increase of the dry/wet ratio with photodegradation conforms to Ifju's (1964) conclusion that the material of shorter cellulose chains or shorter crystalline portions is more affected by water than the material of longer chain structure. The fact that the dry to wet ratio in both spans increases during 7 days of weathering indicates that it is not delignification, but the effect of water on cellulose structures, which is the primary consequence of samples wetting. Furthermore, based on FT-Raman spectra of wood heat treated at low temperatures, Yamauchi *et al.* (2005) concluded that water has little or no contribution to chemical reactions of lignin. However, while dry/wet ratio (Table 3) changes gradually for spruce, pine retains almost constant ratio, while lime was shown to exhibit very intensive changes in this ratio (Derbyshire and Miller, 1981). These differences may be due to the amount and distribution of

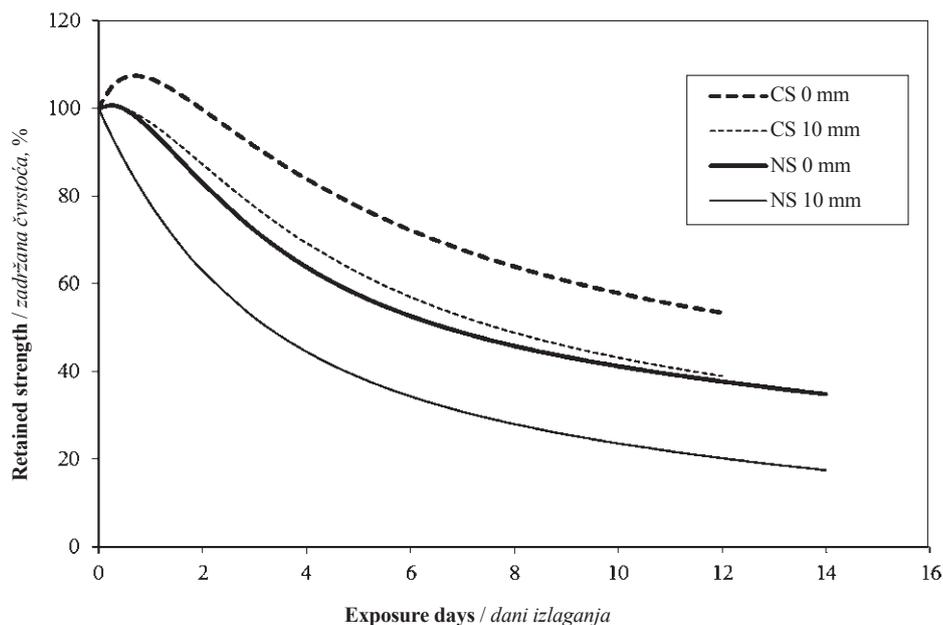
lignin (Derbyshire and Miller, 1981; Agarwal, 2006; Lehringer *et al.*, 2008), the length and crystallinity of cellulose (Ifju, 1964; Newman and Hemmingson, 1990; Andersson *et al.*, 2004), variations in the physical properties and anatomical organization of wood tissue (Wardrop, 1951; Wellwood, 1962; Kennedy, 1966; Nordman and Quickstrom, 1969), or by combinations of these parameters, as proposed by Evans (1984). These aspects should be, however, further addressed in further research.

### 3.2 Effect of density and latewood portion

#### 3.2. Utjecaj gustoće i udjela kasnog drva

Wood density was shown to greatly determine the degradation rates. This is in accordance with the findings by Feist and Mraz (1978) and Arnold *et al.* (1991), who determined that erosion rates during full natural exposure can be significantly changed with only 10 % variations in wood density. Density affects the measured strength inasmuch as the level of recorded strength is significantly different (vertical shift of the curves in Figure 2), and the initial increase in strength noticeably differs for specimens of different density.

However, the density does not seem to affect the rates of strength loss, since the curves of strength changes remain parallel throughout a longer exposure. The wood of higher density shall weather slower, but the deterioration generally develops at the same rate as with woods of



**Figure 2** Strength changes of spruce coded as Croatian spruce (CS, density 480 g/cm<sup>3</sup>) and Norway spruce (NS, density 370 g/cm<sup>3</sup>) during artificial weathering in humid conditions in QUV-2 regime

**Slika 2.** Promjene čvrstoće hrvatske smrekovine (CS, gustoća 480 g/cm<sup>3</sup>) i norveške smrekovine (NS, gustoća 370 g/cm<sup>3</sup>) tijekom laboratorijskog izlaganja u uvjetima visoke vlažnosti QUV-2

lower density, except in initial stage of weathering. After prolonged weathering exposures, wood of different density could reach the same level of degradation.

Interestingly enough, there were cases recorded in this experiment when the density was not a sufficient parameter for the estimation of the tensile properties of the wood material. The aberrations in linearity of the density - tensile strength relationship, which Biblis (1970) defined for tangential earlywood and latewood microtensile specimens, were too small to explain the irregularities recorded in our experiment. Table 1 shows that the ranking of the blocks for sectioning regarding the initial strength of their strips was not directly related to their density, but rather to the proportion of the latewood in the growth rings. That leads to

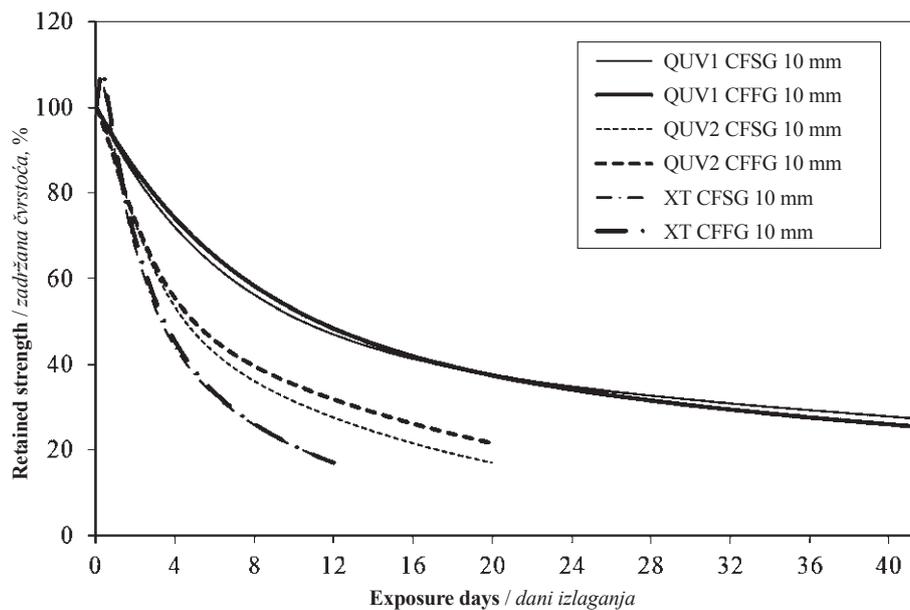
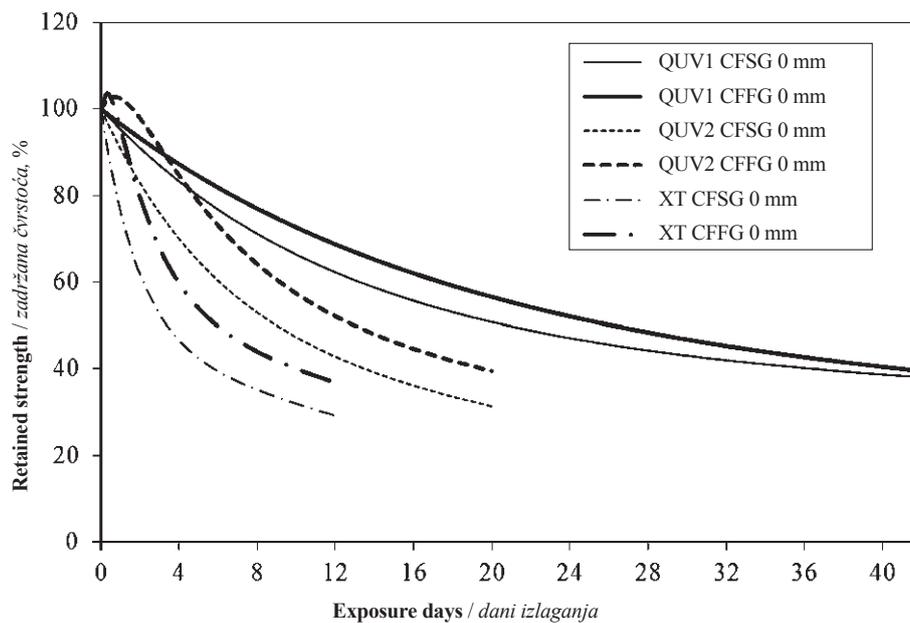
the conclusion that it is earlywood that degrades at faster rate, while denser material will degrade at a slower pace. The reflection of the degradation on tensile properties, however, will depend more on latewood proportion than on density, since latewood controls the recorded changes in tensile strength.

### 3.3 Effect of growth rate

#### 3.3. Utjecaj brzine rasta

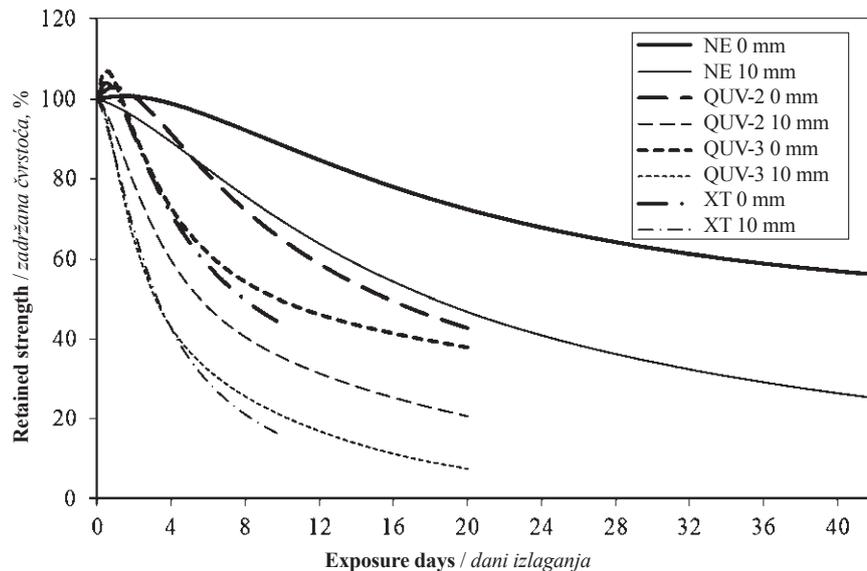
The effect of the rate of growth has been tested using fir specimens of two distinct growth rates designated here as “slow grown” (SG in further text) and “fast grown” (FG) wood.

The visual difference in the growth rate of the two sets of fir blocks was very pronounced, but the



**Figure 3** Strength changes of slow grown (CFSG) and fast grown (CFFG) Croatian fir in different levels of humidity effect in QUV-1, QUV-2 and XT regime

**Slika 3.** Promjene čvrstoće jelovine sporog rasta (CFSG) i brzog rasta (CFFG) tijekom izlaganja različitim uvjetima vlažnosti u režimu QUV-1, QUV-2 i XT



**Figure 4** Strength changes of Scots pine sapwood (SPS) in natural exposure and in artificial weathering regimes of high humidity

**Slika 4.** Promjene čvrstoće bjeljike bijele borovine (SPS) tijekom prirodnoga i laboratorijskih izlaganja pri visokoj relativnoj vlažnosti zraka

density and latewood portions were shown to exhibit certain anomaly. Slow grown material consisted of lower latewood proportion and lower density than “fast grown” strips. Initial strength values did not differ much, especially not in the finite span testing (Table 1). Figure 3 shows that in the finite span testing, the curves of fast and slow grown material show almost perfect match. On the contrary, in zero span testing, the curves never match; the fast grown material degrades at somewhat lower rate. In humid exposure conditions, the fast grown specimens exhibit a shoulder, and this seems to “shift” the curve to the up and right. This never happened with the slow grown material. Even in dry conditions (QUV-1), the fast grown material shows a tendency to strength retention in the initial phase. It is important to notice that the same positioning of the strength loss curves was observed in all regimes.

It seems that the characteristic structural behavior in tension - as seen in finite span testing - is dominated by the species’ anatomical features and does not differ significantly for the given range of density and latewood portion. This is valid for the absolute values of the ultimate load as well as for the rate of its changes with the time of exposure. However, the cellulosic microfibrillar strength – as seen in zero span results – greatly depends on the organization of the cellulosic structural elements in the latewood bands. Zero span testing can depict very fine cellulose changes, but caution must be taken so as not to misjudge the strength changes – e.g. appearance or absence of the shoulder - by mechanical aspects of the testing process.

The reduction in ring width generally leads to the increase in density and consequently to higher mechanical properties. The growth rate should nevertheless be combined with density and latewood proportion to form a set of parameters, which determine the strength level and strength loss rates of particular wood material. This is in accordance with the findings by

Feist and Mraz (1978), who found that fast grown latewood erodes more slowly than slow grown latewood, due to thinner and more fragile cell walls of the slow grown latewood. It must be emphasized that caution must be taken about the density and growth rates not only because of their influence on weathering behavior but also because of the sensitivity and dependence of the tensile testing procedure on these parameters.

### 3.4 Effect of weathering conditions

#### 3.4. Utjecaj uvjeta izlaganja

When strips were exposed to UV light using the same mounting system as for those exposed to Xenon source (QUV-3 and XT curves in Figure 4), there were virtually no differences in the degradation rates between the two machines. Surprisingly enough, the XT curve presents the strength loss of the material of the density (440 kg/m<sup>3</sup> - SPS1, Table 1) lower than the one used in the QUV-3 test (540 kg/m<sup>3</sup> - SPS2, Table 1). This would mean that the degradation effect in the UV is relatively more intensive, for it had caused similar degradation rates of significantly denser material.

The output of the Xenon source with window glass filter in the spectral range 295 - 540 nm is 567 W/m<sup>2</sup> and that of the UVA-340 lamps is 37 W/m<sup>2</sup>, which makes the ratio of 15.3. If consideration of the radiation output is restricted to the wavelength range 295 - 400 nm, i.e. to UV output only, then the output of the Xenon source is 133 W/m<sup>2</sup> and that of the UV remains 37 W/m<sup>2</sup>. Thus the ratio of the UV outputs of the two machines is 3.6, still considerably higher than the acceleration factors recorded from the strength loss curves. As can be seen in Fig. 4, exposure conditions like relative humidity, fluctuations of climatic conditions, thermal effects, etc. can significantly influence the strength changes at the same levels of radiation in a weathering machine. Additionally, Figure 4 reflects the relationship between the effects of different levels of

radiation on the same time basis. The results from natural weathering with its unpredictable and stochastic variations in exposure conditions, is presented only for a better insight into the relationship between various exposure regimes and recorded tensile properties.

It would, therefore, seem that the precise nature of the spectral distribution of the radiation source does not significantly affect the nature of the photodegradation process of wood strips. The degradation rates are enhanced by a high UV content in the radiation spectrum. Since no great differences in the degradation rates were observed in the machines of different intensities even in a narrow 300–400 nm range, it may be postulated that the lower portion of the UV spectrum is responsible for the initiation of photodegradation.

#### 4 CONCLUSION

##### 4. ZAKLJUČAK

The microtensile testing proved to be a sensitive and precise tool in monitoring the alterations of wood composition (due to degradation by light or elements). However, the results clearly showed that specific physical and structural properties of material may have detrimental effect on consistency and coherence of results. Latewood proportion and its tensile strength were shown to dominate the tensile testing process.

The comparisons between various species should be made on the material of average density and latewood proportion for each species, so as not to mix the effect of variations in physical and structural properties with the effects caused by main experimental variable, such as weathering resistance of particular timber species.

Generally, higher density results in greater strength and lower degradation rates of wood material. Based on the experiment shown here, it can be seen that such general conclusion must be taken with caution. Its latewood proportion and its mechanical properties affect both the degradation rates and tensile testing reliability.

The exposures in both artificial devices (Xenon and UV source) were shown to offer a satisfactory range of conditions to enable the testing of weathering degradation rates of wood. Despite the great differences in the spectral distribution of the radiation sources, the results differed only in the rate of degradation. The fact that strength loss changes followed the same pattern indicates that there is no difference in the nature of degradation process in various weathering machines, but the speed and rates of degradation may be different. Careful choice of material and artificial weathering conditions forms a basis for the sound comparison between the artificial and natural weathering regimes.

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#### 5 REFERENCES

##### 5. LITERATURA

- Andersson, S.; Wikberg, H.; Pesonen, E.; Maunu, S. L.; Serimaa, R., 2004: Studies of crystallinity of Scots pine and Norway spruce cellulose. *Trees*, 18: 346-353. <https://doi.org/10.1007/s00468-003-0312-9>.
- Agarwal, U. P., 2006: Raman imaging to investigate ultrastructure and composition of plant cell walls: distribution of lignin and cellulose in black spruce wood (*Picea mariana*). *Planta*, 224: 1141-1153. <https://doi.org/10.1007/s00425-006-0295-z>.
- Altgen, M.; Militz, H., 2016: Photodegradation of thermally-modified Scots pine and Norway spruce investigated on thin micro-veneers. *European Journal of Wood and Wood Products*, 74: 185-190. <https://doi.org/10.1007/s00107-015-0980-3>.
- Arnold, M.; Feist, W. C.; Williams, R. S., 1991: Effect of weathering of new wood on the subsequent performance of semitransparent stains. *Forest Products Journal*, 42: 10-14.
- Biblis, E. J., 1970: Effect of thickness of microtome sections on their tensile properties. *Wood and Fiber Science*, 2: 19-30.
- Bischof Vukušić, S.; Katović, D.; Schramm, C.; Trajković, J.; Šefc, B., 2006: Polycarboxylic acids as non-formaldehyde anti-swelling agents for wood. *Holzforchung*, 60: 439-444.
- Derbyshire, H.; Miller, E. R., 1981: The photodegradation of wood during solar irradiation. Part 1. Effects on the structural integrity of thin wood strips. *Holz als Roh- und Werkstoff*, 39: 341-350. <https://doi.org/10.1007/BF02608404>.
- Derbyshire, H.; Miller, E. R.; Turkulin, H., 1995: Investigations into the photodegradation of wood using microtensile testing. Part 1: The application of microtensile testing to measurement of photodegradation rates. *Holz als Roh- und Werkstoff*, 53: 339-345. <https://doi.org/10.1007/s001070050103>.
- Derbyshire, H.; Miller, E. R.; Turkulin, H., 1996: 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. *Holz als Roh- und Werkstoff*, 54: 1-6. <https://doi.org/10.1007/s001070050123>.
- Derbyshire, H.; Miller, E. R.; Turkulin, H., 1997: Investigations into the photodegradation of wood using microtensile testing. Part 3: The influence of temperature on photodegradation rates. *Holz als Roh- und Werkstoff*, 55 (5): 287-291. <https://doi.org/10.1007/s001070050229>.
- Evans, P. D., 1984: Physical and biological factors affecting the weathering of wood surfaces. Ph. D. Thesis. University of Wales, Bangor.
- Feist, W. C.; Mraz, E. A., 1978: Comparison of outdoor and accelerated weathering of unprotected softwoods. *Forest Products Journal*, 28 (3): 38-43.
- Evans, P. D.; Wallis, A. F. A.; Owen, N. L., 2000: Weathering of chemically modified wood surfaces. Natural weathering of Scots pine acetylated to different weight gains. *Wood Science and Technology*, 34: 151-165. <https://doi.org/10.1007/s002260000>.
- Ifju, G., 1964: Tensile strength behavior as a function of cellulose in wood. *Forest Products Journal*, 21: 366-372.

15. Jirouš-Rajković, V.; Turkulin, H.; Miller, E. R., 2004: Depth profile of UV-induced wood surface degradation. *Surface coatings international. Part B, Coatings transactions*, 87 (4): 235-308.
16. Kataoka, Y.; Kiguchi, M.; Fujiwara, T.; Evans, P. D., 2005: The effects of within-species and between-species variation in wood density on the photodegradation depth profiles of sugi (*Cryptomeria japonica*) and hinoki (*Chamaecyparis obtusa*). *Journal of Wood Science*, 51:531-536 <https://doi.org/10.1007/s10086-004-0685-4>.
17. Kennedy, R. W., 1966: Intra-increment variation and heritability of specific gravity, parallel-to-grain tensile strength, stiffness and tracheid length in clonal Norway spruce. *Tappi Journal*, 49: 292-296.
18. Klüppel, A.; Mai, C., 2017: Effect of seawater wetting on the weathering of wood. *European Journal of Wood and Wood Products*. (first online) <https://doi.org/10.1007/s00107-017-1268-6>.
19. Lehringer, C.; Gierlinger, N.; Koch, G., 2008: Topochemical investigation on tension wood fibres of *Acer* spp., *Fagus sylvatica* L. and *Quercus robur* L. *Holzforchung*, 62: 255-263. <https://doi.org/10.1515/HF.2008.036>.
20. Lehringer, C.; Saake, B.; Živković, V.; Richter, K.; Militz, H., 2011: Effect of *Physisporinus vitreus* on wood properties of Norway spruce. Part 2: Aspects of microtensile strength and chemical changes. *Holzforchung*, 65: 721-727. <https://doi.org/10.1515/hf.2011.090>.
21. Newman, R. H.; Hemmingson, J. A., 1990: Determination of the degree of cellulose crystallinity in wood by carbon-13 nuclear magnetic resonance spectroscopy. *Holzforchung*, 44: 351-355. <https://doi.org/10.1515/hfsg.1990.44.5.351>.
22. Nordman, L. S.; Quickström, B., 1969: Variability of the mechanical properties of fibers within a growth period. In: D. H. Page (ed.): *The physics and chemistry of wood pulp fibers*. TAPPI STAP Series, No. 8. New York: TAPPI, p. 177-201.
23. Turkulin, H.; Sell, J., 2002: Investigations into the photodegradation of wood using microtensile testing. Part 4: Tensile properties and factography of weathered wood. *Holz Roh- Werkstoff*, 60: 96-105. <http://dx.doi.org/10.1007/s00107-002-0282-4>.
24. Turkulin, H.; Derbyshire, H.; Miller, E. R., 2004: Investigations into the photodegradation of wood using microtensile testing. Part 5: The influence of moisture on photodegradation rates. *Holz als Roh- und Werkstoff*, 62: 307-312. <https://doi.org/10.1007/s00107-004-0493-y>.
25. Wardrop, A. B., 1951: Cell wall organization and the properties of the xylem. 1. Cell wall organization and the variation of breaking load in tension of the xylem in conifer stems. *Australian Journal of Science and Research B*, 4: 391-414.
26. Wellwood, R. W., 1962: Tensile testing of small wood samples. *Pulp Paper Mag. Can.* 63:T61 – T67.
27. Xie, Y.; Krause, A.; Militz, H.; Turkulin, H.; Richter, K.; Mai, C., 2007: Effect of treatments with 1, 3-dimethyl-4, 5-dihydroxy-ethyleneurea (DMDHEU) on the tensile properties of wood. *Holzforchung*, 61: 43-50. <http://dx.doi.org/10.1515/HF.2007.008>.
28. Yamauchi, S.; Iijima, Y.; Doi, S., 2005: Spectrochemical characterization by FT-Raman spectroscopy of wood heat treated at low temperatures: Japanese larch and beech. *Journal of Wood Science*, 51: 498-506. <http://dx.doi.org/10.1007/s10086-004-0691-6>.
29. Živković, V.; Turkulin, H., 2014: Microtensile testing of wood – overview of practical aspects of methodology. *Drvna industrija*, 65 (1): 59-70. <http://dx.doi.org/10.5552/drind.2014.1320>.
30. Živković, V.; Arnold, M.; Richter, K.; Radmanović, K.; Turkulin, H., 2014: Spectral sensitivity in the photodegradation of fir wood (*Abies alba* Mill.) surfaces: colour changes in natural weathering. *Wood Science and Technology*, 48: 239-252. <https://doi.org/10.1007/s00226-013-0601-4>.
31. Živković, V.; Arnold, M.; Pandey, K. K.; Richter, K.; Turkulin, H., 2016: Spectral sensitivity in the photodegradation of fir wood (*Abies alba* Mill.) surfaces: correspondence of physical and chemical changes in natural weathering. *Wood Science and Technology*, 50: 989-1002. <https://doi.org/10.1007/s00226-016-0834-0>.

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