

Article

Influence of Ageing on Abrasion Volume Loss, Density, and Structural Components of Subfossil Oak

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Abstract: Subfossil oak wood has spent centuries or millennia in the aquatic medium (rivers, lakes, bogs, etc.) and, due to water anoxic conditions, its decomposition is very slow. As a result of its long residing in specific conditions, its chemical composition, appearance, as well as mechanical and tribological properties have changed. Because of its aesthetic and mechanical properties, subfossil wood is very attractive and often used to produce valuable objects. The main objective of this study was to test how abrasion wear resistance of subfossil oak is affected by ageing. The effects of ageing on wood density and on the structure of lignin and cellulose were tested, as well as the loss of volume during abrasion in correlation with these changes. A study was conducted on samples of recent (regular) pedunculate oak wood and on six subfossil pedunculate oak samples in the age range of 890 and nearly 6000 years. Abrasion wear resistance was expressed through the loss of volume recorded using the Taber abraser. The smallest abrasion volume loss was measured for the recent oak specimens. Linear regression analyses showed that there was a very strong negative linear relationship between the age of subfossil oak and its abrasion volume loss. There was also a strong, but positive and significant linear correlation between subfossil oak age and density. Ageing also affected the structural composition of wood. Results obtained by Fourier Transform Infrared spectroscopy indicated a reduction of the relative crystalline fraction of subfossil wood in recent oak. The degradation of lignin in subfossil oak samples progressed more slowly over time than cellulose degradation. There was a negative correlation between age and the ratio of cellulose and lignin degradation; however, that relationship was found statistically insignificant. Similar results were obtained for the relationship between abrasion wear resistance and changes in the structural composition of the studied samples of subfossil oak wood.

Keywords: subfossil wood; oak; abrasion wear resistance; FTIR analysis; density



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1. Introduction

Wet and anaerobic conditions that exist in riverbeds, bogs, peatlands, and similar aquatic areas may preserve wood for many hundreds and even thousands of years [1,2]. In such habitats, wood biodeterioration is very slow and mainly bacterial, because typical terrestrial wood degraders (insects, calms, brown rot, white-rot fungi, etc.) needoxic conditions for their growth. Based on the micromorphological degradation pattern they produce, microorganisms categorized as erosion bacteria are the most common involved in microbiological attack in anaerobic aquatic environments [3]. The wood found and preserved in waterlogged conditions is called abonos, subfossil wood, or bog wood. Numerous wood species (e.g., oak, elm, and ash) can be preserved that way. Among these species, oak, in particular, is preserved exceptionally well. The good preservation of old oak trunks may be the result of a high content of tannins, which also inhibit the deleterious

activity of bacteria and fungi. Due to mineral deposition from river water into the wood, the durability, appearance, and structure of the wood are modified [4–6]. Subfossil oak is highly appreciated for its hardness and durability as well for its attractive dark color and good acoustic properties and is therefore often used for the production of musical instruments, ornaments, jewelry, sculptures, and also for luxurious furniture, parquet, and veneers [2,7,8].

Various physical and chemical properties of subfossil wood have been investigated in Croatia [2,9–11] and abroad [12–16], but in the literature, we did not find any data concerning the measurement of abrasion resistance (or of resistance to any other wear mechanisms) in subfossil oak wood. Abrasive wear resistance can be defined as the ability to resist mechanical wear caused by hard particles that move along a solid surface (ASTM G40-21a) [17]. This property depends on the hardness, yield strength, and density of wood and on the abrasive grain size, but it is not possible to set up an entirely unambiguous quantitative relationship between these properties [13,18,19].

In general, the analysis of the mechanical, physical, and chemical properties of subfossil oak shows that the properties of subfossil wood have changed in relation to those of recent oak. However, these changes caused by time as well as the conclusions reached by different examiners have not always been consistent. The reason for that is the different aquatic conditions in which the examined samples originated [7,20–24].

Ordinarily, the composition of wood includes three elements, i.e., cellulose, lignin, and hemicellulose. Previous studies showed that with ageing, the wood content of cellulose decreases, and that of lignin increases in relation to the composition of recent wood [21]. Cellulose is a crystalline fraction, while lignin and hemicellulose are amorphous fractions in wood. Wood crystallinity may be a suitable indicator for estimating the level of wood degradation during ageing. Crystallinity also strongly affects the tribological, physical, mechanical, and chemical properties of wood. FTIR spectroscopy is one of the methods that can provide details about the crystallinity and structural characteristics of wood and, consequently, can give useful information about wood degradation [12,25–27].

This paper presents the results of abrasion wear resistance measurements for recent (regular) oak samples and six subfossil oak samples which were collected in Northern Bosnia and Herzegovina. Since the abrasion resistance of subfossil oak has not been studied yet and is an important property for machining (subfossil wood often causes a strong blunting of tools), the goal of the present research was to summarize and analyze the possible correlation between abrasion wear resistance, density, and changes found in wood structural components caused by ageing in aquatic medium.

2. Materials and Methods

Six subfossil oak trunks (samples 1–5) were retrieved from the Sava riverbed between the villages of Grebnice (45.046648, 18.528779) and Domaljevac (45.074475, 18.588781) in Bosnia and Herzegovina (Figure 1). The trunks were found under a 1–2 m layer of gravel in the central part of the riverbed where the depth of the river is never less than 5–6 m. The oldest subfossil oak trunk (sample 6) originated from a gravel pit in the village of Oštra Luka (45.001281, 18.568225). That gravel pit developed following the gravel exploitation within the area of the Sava River branch. The trunk was taken from a depth of 8 to 9 m under the water level of the lake, below a thin layer of sand and silt.

Sample 0 was regular recent pedunculate oak wood from an approximately 70-year-old tree, stored indoors for 5 years. That trunk (sample 0) originated from the forest near the village of Štitar (45.107301, 18.632835), approximately 5 km from the location where the first five subfossil trunks were retrieved (Figure 1). Its growth ring width was about 1.6 mm.

This part of the land is characterized by a lowland fluvial accumulation-type of relief, slightly sloping towards the east, and is dominated by alluvial calcareous loamy soils (Figure 1). The samples of subfossil wood were dated by means of the Carbon-14 dating method. The ages and locations of the subfossil samples are listed in Table 1.

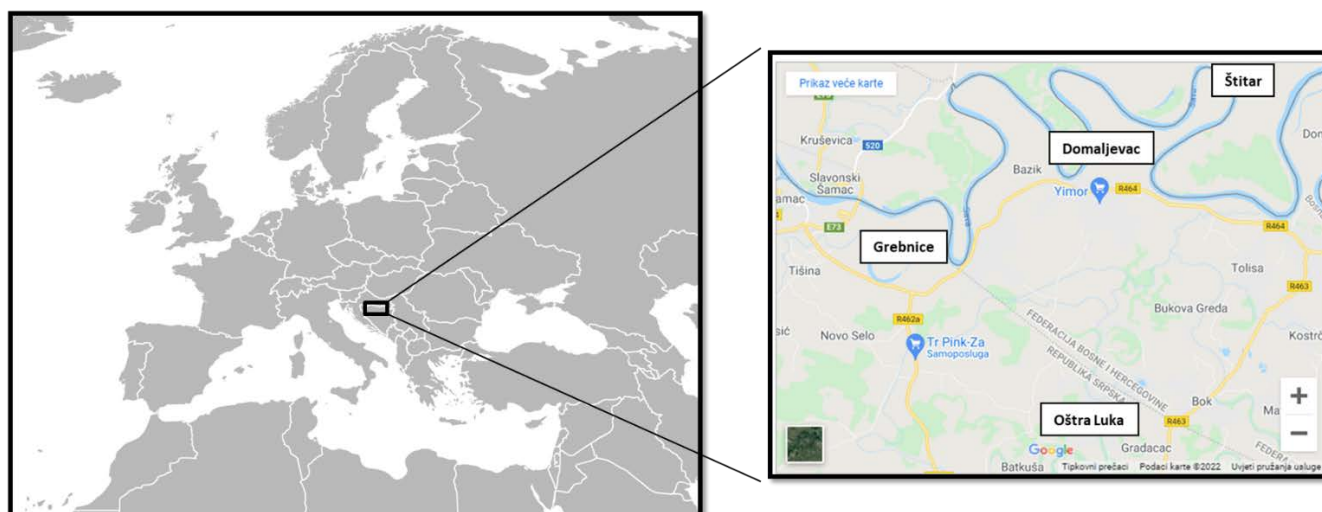


Figure 1. View of the oak samples' locations of origin [28].

Table 1. The origin of the examined subfossil samples and their age.

ID Sample	Origin of Sample	Conventional ^{14}C Age (BP)/Years
1	Grebnice/Domaljevac	890 ± 50
2	Grebnice/Domaljevac	1195 ± 50
3	Grebnice/Domaljevac	1840 ± 55
4	Grebnice/Domaljevac	3890 ± 65
5	Grebnice/Domaljevac	4555 ± 65
6	Oštra Luka	5890 ± 115

Microstructure analysis, abrasion wear resistance testing, density measurements, moisture content measurements, and FTIR analysis were performed on all samples, from 0 to 6.

The wood specimens intended for microstructural determination analyses were prepared as thin cross sections with a microtome and were examined under an OLYMPUS BX 51-P transmittance light microscope for the purpose of wood taxa identification. The wood anatomy atlas [29] was used for the identification of the examined samples. The test pieces intended for the determination of abrasion wear resistance were cut from the heartwood, their dimensions being $5 \text{ mm} \times 5 \text{ mm} \times 40 \text{ mm}$ (Figure 2). All pieces were acclimatized for a few weeks at room temperature. Wood specimens showing cracks or some other defects were rejected and not used in the test. Ten wood specimens were examined for each sample (0–6). The length of all test pieces (40 mm) coincided with the natural axes of fiber orientation (longitudinal direction), and the friction surface ($5 \text{ mm} \times 5 \text{ mm}$) coincided with the cross section of the wood. We selected the test specimens from oak trunks with an as similar as possible growth ring width (1.3–1.9 mm), to avoid the influence of the percentage of late and early wood on abrasion wear resistance [30]. The samples of subfossil oak were much darker than the sample of recent oak (Figure 2), owing to the reaction between the iron from the riverbed soil and the tannins in the oak wood [7].

For the evaluation of abrasion wear resistance, a Taber abraser with a 125 mm rotating abrasive disc was used (Figure 3). The disc rotational speed was 1 rev/s, and the load applied to keep the test pieces in contact with sandpaper was 4.91 N. The sandpaper was designated as P180, with an $82 \mu\text{m}$ mean diameter of the abrasive particles. To remove sawdust during the process of abrasion, a vacuum cleaner was used. The weight loss (Δm) of the test pieces was measured after 300 grinding cycles. According to the ISO 13061-1:2014 standard [31], the moisture content of the samples was determined in order to calculate the mass loss (Δm_{12}) and density (ρ_{12}) at 12% moisture content. The density ρ_{12} was calculated according to ISO 13061-2:2014 standard [32]. The mass loss (Δm_{12}) was converted into

the abrasion volume loss (ΔV_{12}). The abrasive wear resistance was expressed as the value inversely proportional to the volume loss value.



Figure 2. Wood specimen prepared for the abrasion testing arranged from the recent oak (0) to the oldest subfossil oak (1–6).

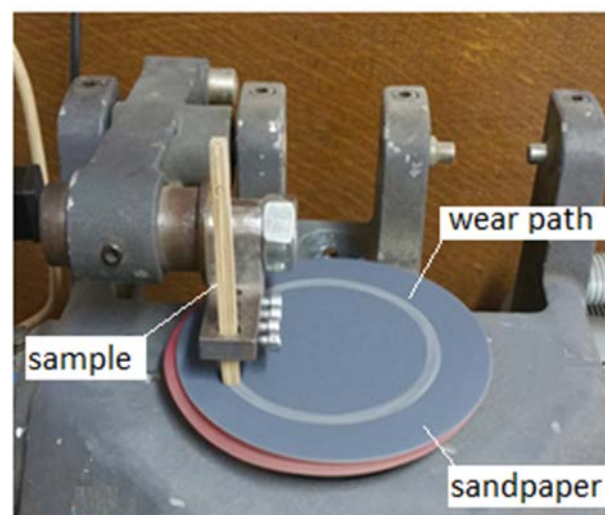


Figure 3. Taber abraser.

Fourier Transform Infrared (FTIR) analysis was performed by an IR Spirit spectrometer (Shimadzu, Japan) equipped with an ATR device with a single-reflection diamond crystal. The spectra were collected in the range from 4000 to 400 cm^{-1} over 32 scans at a resolution of 2 cm^{-1} . The FTIR-ATR analysis was done directly on the surface of the wood samples (three replicates) at room temperature. The normalized intensities of all peaks were calculated using the intensity of the reference band at 1031 cm^{-1} .

We performed linear regression analyses to define the relationships between abrasion volume loss, age of the samples, density, and degree of degradation. The statistical analysis was performed in Microsoft Excel, version 2102.

3. Results and Discussion

The microstructural determination analyses showed that all the subfossil trunks examined in this study belonged to the subgenus *Quercus* (Figure 4). Although the species of the subgenus *Quercus* cannot be distinguished reliably from one another only on the basis of their wood anatomy [29], due to the ecology of few *Quercus* species that grow in the Sava River Basin [33–35], we are confident that all the tested samples originated from the *Quercus robur* species (pedunculate oak).

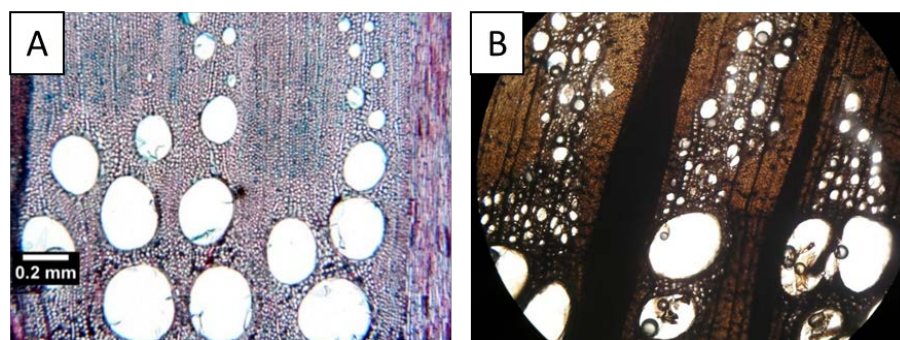


Figure 4. Microstructure of (A) recent normal wood and (B) subfossil (sample 6) oak wood.

Table 2 shows the results of the measurements of mass loss after 300 grinding cycles and of density at 12 % moisture content.

Table 2. Mass loss after 300 grinding cycles and density at 12% moisture content (MC), I_{1368}/I_{1509} and I_{1319}/I_{1336} of oak wood samples (X_p , mean value, SD , standard deviation, CV , coefficient of variation (%), min, minimum value, max, maximum value).

SAMPLE ID	Δm_{12} (g)				ρ_{12} (g/cm ³)					I_{1368}/I_{1509}	I_{1319}/I_{1336}
	X_p	SD	Min	Max	X_p	SD	Min	Max	CV		
0	0.067	0.003	0.062	0.070	0.798	0.028	0.760	0.833	3.5	1.22	1.08
1	0.082	0.007	0.072	0.094	0.714	0.022	0.685	0.742	2.9	1.20	0.88
2	0.081	0.004	0.077	0.085	0.725	0.049	0.653	0.794	6.7	0.84	0.83
3	0.075	0.004	0.071	0.083	0.749	0.025	0.706	0.803	3.3	0.46	0.85
4	0.073	0.005	0.064	0.080	0.733	0.064	0.652	0.821	8.7	0.56	0.82
5	0.073	0.006	0.066	0.084	0.773	0.026	0.729	0.802	3.4	0.67	0.94
6	0.071	0.005	0.065	0.079	0.802	0.013	0.780	0.821	1.6	0.68	0.84

The mean values of volume loss of the samples ranged from 83.5 to 116.9 mm³. The normal wood sample (0) showed the lowest value of volume loss. All subfossil oak samples (1–6) showed a higher value of volume loss and lower abrasive wear resistance compared to the recent oak (0) (Figure 5). If only the subfossil oak samples were taken into consideration, linear regression analyses of the test results indicated that there was a strong negative linear relationship between abrasive volume loss and age (Figure 6). The correlation coefficient for this relationship was -0.927 ($R^2 = 0.859$). This decrease of abrasive volume loss could be unexpected, because of the presumption that with age, abrasion resistance will continue decreasing. Although the degradation of wood progresses with time, simultaneously, the process of fossilization occurs, and the content of iron, calcium, ash, and extractives in oak wood increases [7,21,22]. The process of fossilization is the most probable microstructural reason for abrasion wear resistance growth. Although there is a strong negative linear relationship between abrasive volume loss and age, we must be aware that some previous investigations showed that for subfossil samples, the time factor was of minor importance, while the environment played the main role in the process of destruction and fossilization of wood as well as in the changing of its mechanical and physical properties. An example is reported in the study of Kolar and Rybníček [20] of the physical and mechanical properties

of sub-fossil oak and their comparison with those of recent wood. The research showed an unambiguous difference between a recent sample and all subfossil samples, but the properties of subfossil samples were not clearly dependent on the age of the samples. Five of the subfossil samples investigated in this study originated from a very short section of the river Sava (Figure 1); therefore, in this study, the quality of water and the geological background of the test specimens were almost the same, which certainly influenced by such a strong relationship between age and abrasion wear resistance.

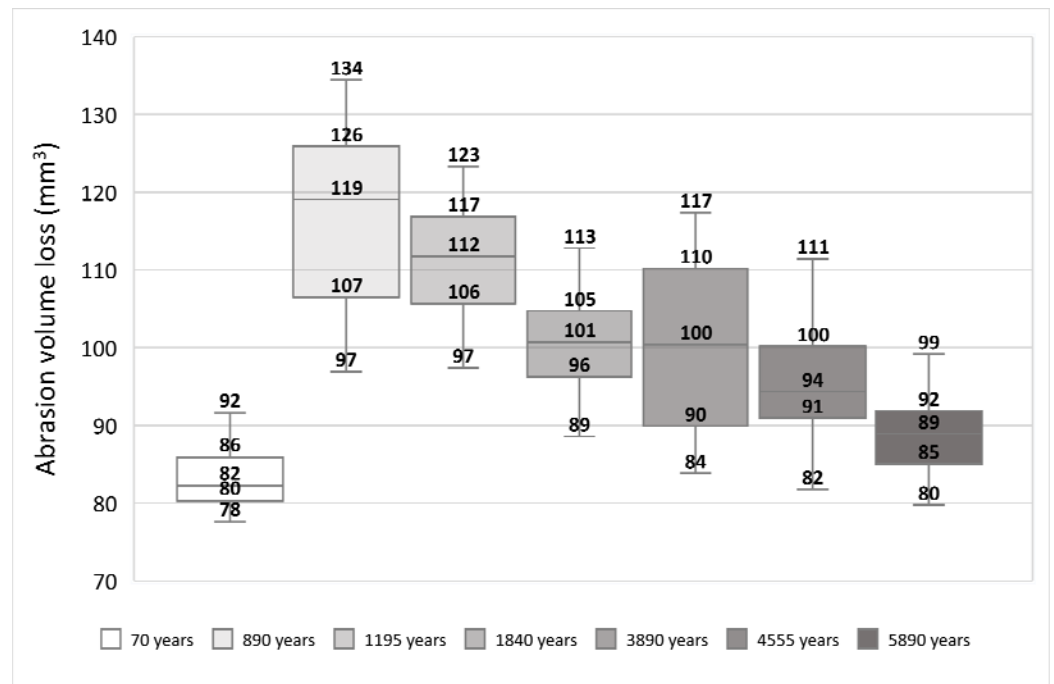


Figure 5. Abrasion volume loss of recent (white) and subfossil oak wood samples (gray).

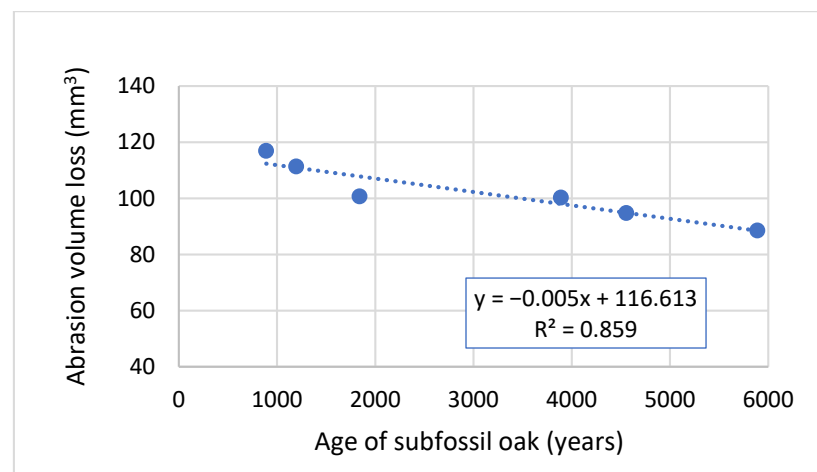


Figure 6. Correlation between abrasion volume loss and age of subfossil oak wood (samples 1–6).

The mean density value of the samples at 12% MC (ρ_{12}) ranged from 0.714 to 0.802 g/cm³ (Table 2). The coefficient of variation for each sample was smaller than 10 %, which according to ISO 13061-2, indicated allowed variability. Figure 7 shows a strong positive linear relationship between the values of subfossil sample density (ρ_{12}) and their age, with correlation coefficient $R = 0.876$ ($R^2 = 0.767$). All subfossil oak specimens of age up to 3890 years had similar density values (0.714–0.733 g/cm³) which were significantly lower than the density values of recent oak (0.798 g/cm³). The two oldest samples of subfossil

oak aged 4555 and 5890 years had a higher density than younger specimens. The density of the oldest subfossil oak (0.802 g/cm^3) was slightly higher than the density of recent oak.

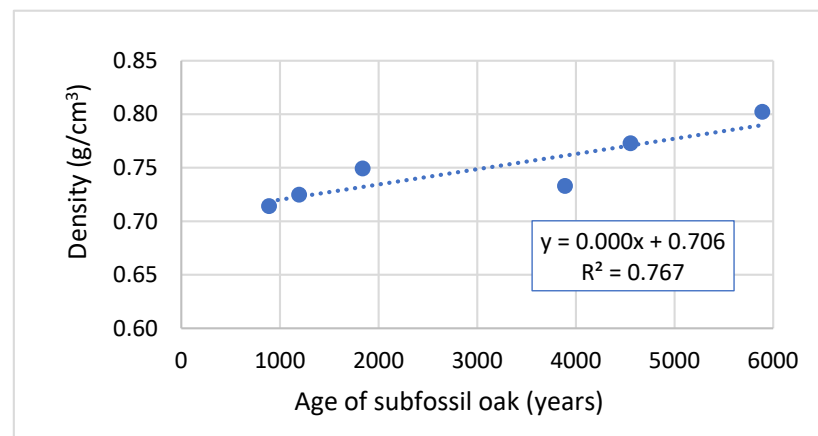


Figure 7. Correlation of density at 12% MC and age of subfossil oak wood samples (1–6).

The decrease in density of subfossil wood compared to recent wood conforms to the results published in the studies by Kolar et al. [20] and Guyette et al. [36]. These studies pointed out that wood density generally decreases with the age. However, a large variation of the results was also noted, and the analyzed samples were younger than the two oldest samples of this study. The increase in density of the two analyzed oldest samples (5 and 6) could be related to mineral deposition from the river water, which was also proved by their dark color, resulting from the high concentration of iron in the wood [7,22]. According to Mankowsky et al. [37], archaeological wood is characterized by a directly proportional relationship between wood density and the content of mineral compounds.

Linear regression analyses showed a strong negative linear correlation, with correlation coefficient $R = -0.948$ ($R^2 = 0.900$), between abrasion volume loss and wood density at 12% MC (Figure 8). The oldest subfossil sample and the normal wood sample (0) showed the highest density values and the lowest volume loss in the abrasion test. Although the subfossil sample had a slightly higher density than the recent oak sample, its volume loss was higher. This conforms to the results obtained in the study of Mankowsky et al. [37], where it was shown that with the same density values, the normal wood (recent) sample had better mechanical properties than the subfossil sample.

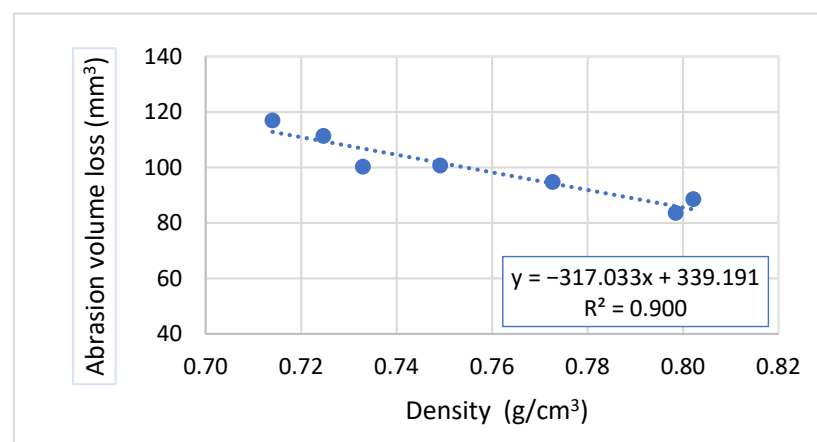


Figure 8. Correlation between abrasion volume loss and density at 12% MC of oak woods samples (0–6).

The chemical composition of the representative control specimen (0) and specimens 1–6 was analyzed by infrared spectroscopy. We recorded the FTIR spectra of the representative

control specimen (0) and specimens 1–6, and all the detected bands are presented in Figure 9. The results for the tested samples (1–6) were compared to the spectra of the representative control specimen (0). The main bands associated with cellulose and hemicelluloses were identified at 3650–3000, 2903, 1736, 1458, 1423, 1368, 1319, 1156, 1103, 1031, and 897 cm^{-1} , while lignin was identified by the bands at 1595, 1509, 1265, and 1232 cm^{-1} [20,27,38–40].

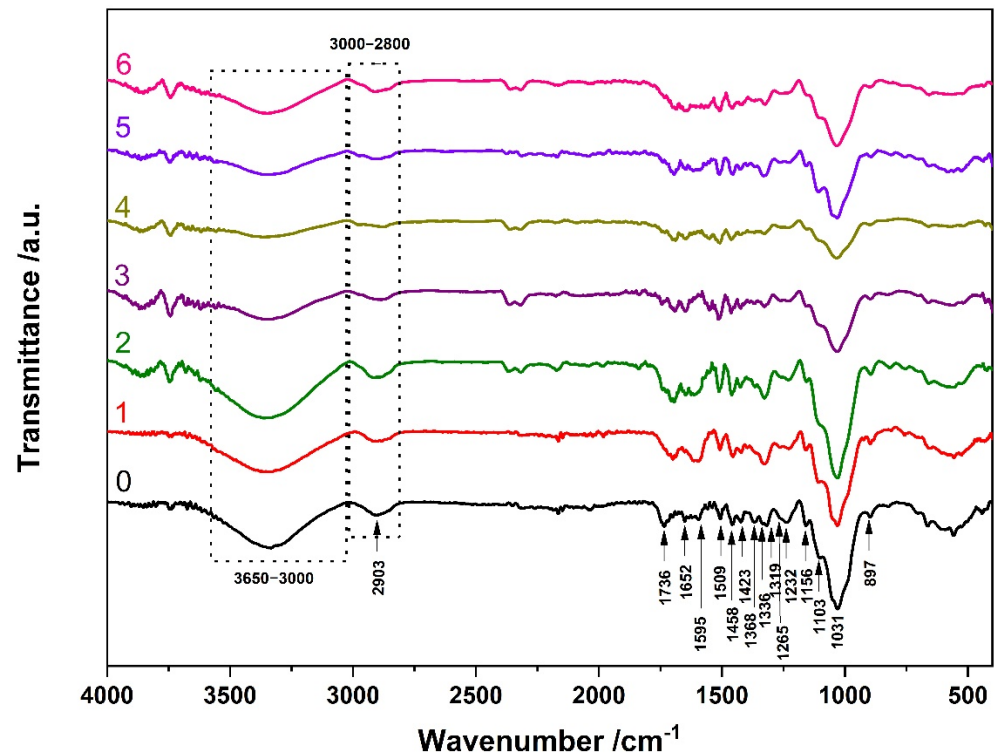


Figure 9. FTIR spectra acquired from wood samples (0–6).

According to the obtained FTIR spectra results, it was found that ageing played an important role in wood structural composition; the intensity of the bands was lower compared to the band intensity of the control specimen (0) (Figure 9).

More useful information on wood degradation with respect to ageing can be obtained by calculating the ratio between bands located at 1368 and 1509 cm^{-1} and at 1319 and 1336 cm^{-1} [27]. The calculated transmittance ratios I_{1368}/I_{1509} and I_{1319}/I_{1336} are shown in Table 2.

The transmittance ratio I_{1368}/I_{1509} can be used as the crystallinity index of wood samples [27]. The degradation fraction of wood can be obtained from the transmittance ratio between the peak at 1368 cm^{-1} , which is attributed to the cellulose fraction, and the peak at 1509 cm^{-1} , attributed to the lignin fraction.

It was found that there was no statistically significant linear correlation between the degree of degradation of wood structural components and the age of the samples, as well as between the abrasion volume loss and the degree of wood structure degradation.

The I_{1368}/I_{1509} ratio decreased during wood ageing compared to the control sample (0) (Table 2). This suggests that the degradation of lignin in subfossil oak samples progressed more slowly over time than cellulose degradation. A linear correlation between the I_{1368}/I_{1509} ratio and wood age gave only a moderate correlation coefficient $R = -0.640$ ($R^2 = 0.410$), Figure 10.

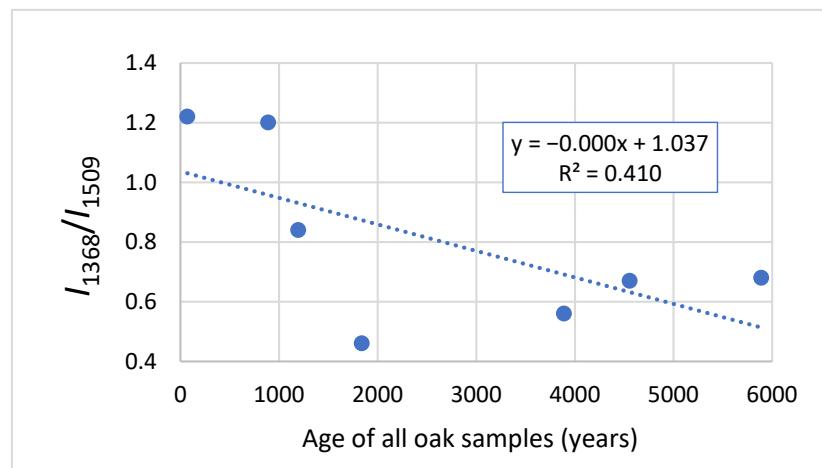


Figure 10. Correlation between the I_{1368}/I_{1509} ratio and wood age of oak wood samples.

As the samples were not exposed over time to the same environmental conditions, such as the presence of oxygen that influences lignin degradation, there appeared a big difference regarding lignin degradation in samples 1–6, as expected. The I_{1368}/I_{1509} ratio had no influence on the loss of volume during abrasion (Figure 11).

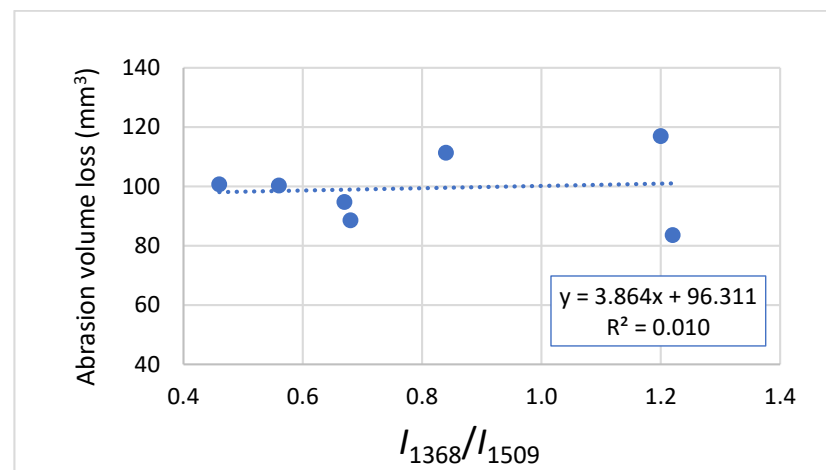


Figure 11. Correlation between abrasion volume loss and the I_{1368}/I_{1509} ratio.

The transmittance ratio between the peaks located at 1319 and 1336 cm^{-1} may give information about the degradation of crystalline and amorphous cellulose. If the I_{1319}/I_{1336} ratio decreases, the degradation of crystalline cellulose is more intense than the degradation of amorphous cellulose [25–27]. The obtained results reported in Table 2 and Figure 12 indicated that in all subfossil samples, the I_{1319}/I_{1336} ratio decreased compared to that for the control sample (0). It can be noted that the ratio was constant for all subfossil samples, except for sample 5, where the degradation of crystalline cellulose was slightly slower than for other samples compared to that in the recent sample. The results obtained indicated that the ageing of subfossil wood has a moderate negative linear relationship with the I_{1319}/I_{1336} ratio, $R = -0.421$ ($R^2 = 0.177$). We also found a moderate negative linear correlation between the I_{1319}/I_{1336} ratio and volume loss during abrasion, with a correlation coefficient of $R = -0.564$ ($R^2 = 0.318$), as shown in Figure 13.

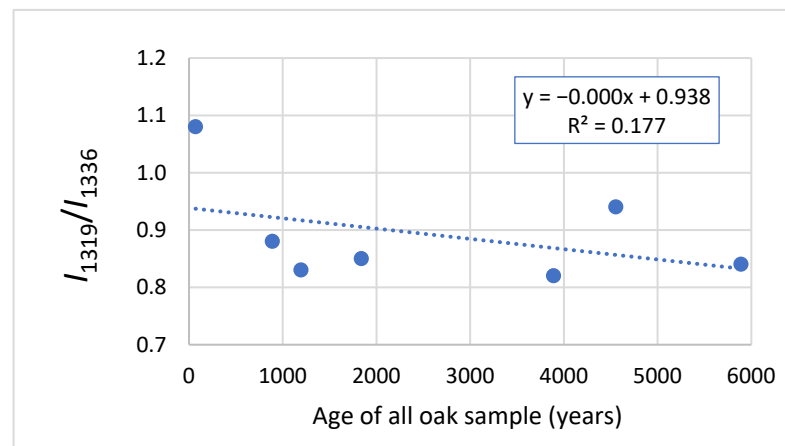


Figure 12. Correlation between the I_{1319}/I_{1336} ratio and the age of oak wood samples (0–6).

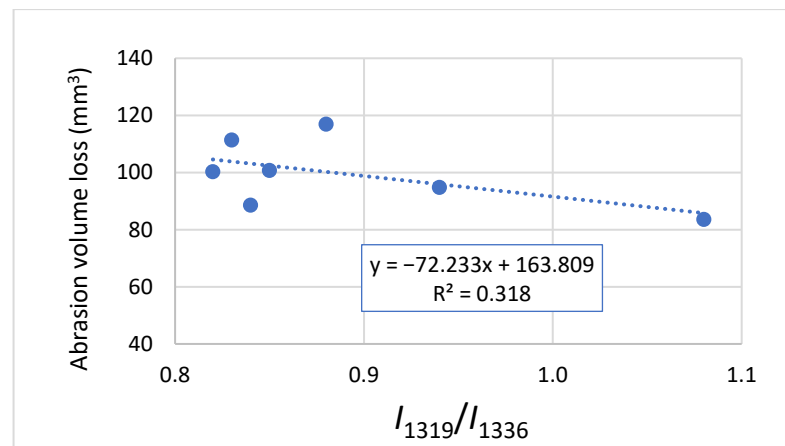


Figure 13. Correlation between abrasion volume loss and the I_{1319}/I_{1336} ratio for samples 0–6.

4. Conclusions

The age of subfossil wood has a great influence on abrasion wear. The recent (normal wood) oak sample showed the highest abrasion wear resistance; with increasing age, the abrasion volume loss of subfossil wood decreased.

There was a strong positive linear correlation between the subfossil oak wood density and its age. The subfossil samples aged up to 3890 years showed a similar density that was much lower than the density of the recent oak. A further increase in age led to a significant increase in the density of subfossil wood.

We found a strong negative linear correlation between abrasion volume loss and wood density.

Lignin degradation compared to cellulose degradation increased more slowly with the increasing age of subfossil oak. Linear regression analyses showed that there was no statistically significant correlation between wood's age and the ratio of cellulose and lignin degradation.

The results showed no linear correlation between abrasion wear resistance and the ratio of lignin and cellulose degradation of subfossil oaks.

Compared to the recent sample, all subfossil oak samples showed a lower ratio of the contents of crystalline and amorphous cellulose. This suggests that the degradation of amorphous cellulose in subfossil oak was slower than the degradation of crystalline cellulose, but there was no significant correlation between them.

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