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A NEW OPPORTUNITY FOR RESEARCH IN ROMANIA – SUBFOSSIL WOOD

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Abstract: *The present paper is a literature review related to subfossil wood, which aims at acquiring knowledge and understanding of the material. The study presents methods for old wood chronology and some properties such as: structural, chemical, physical, and mechanical, compared with recent wood. The results are very useful for the wood industry and will open new paths for the research of this material.*

Key words: *subfossil wood, oak, pine, chronology, chemical composition, properties.*

1. Introduction

In the literature of the last decade, subfossil wood has been a topic for numerous studies, opening up new opportunities for researchers and the wood industry.

In Europe, this material is known and used in applications of interior design and furniture, and it is very popular in the manufacture of small wooden products [35].

In Romania, sufficient information about subfossil wood has not been available until now. Even though some research and projects on this topic have been carried out recently, most of them addressed the dating of the wood and its geographical distribution in certain areas of the country [14], [25], as well as the fluvial geomorphology combined with subfossil wood dendrochronology [25]. Actually, subfossil oak has been reported as a frequent species in the Carpathian region, expanding since 10,200-9500 BP [14]. A new study with Romanian contribution, necessary for the conservation process of the artefacts, referred to the chemical composition of historical oak wood from the fourteenth century [10]. The chemical composition of wood originated from urban pavement from Iaşi *versus* recent wood was investigated.

Over the last few years, many subfossil trees have been discovered buried in various sediments along rivers in Romania, during excavation works. Most of them

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were oak trees. Unfortunately, this wood does not preserve well after digging and a significant proportion of it has been lost, mostly due to the lack of knowledge and information in this field.

Figure 1 presents subfossil oak *in situ* or after excavation in different regions of the country. The samples collected were brought to the Department of Wood Processing and Design of Wooden Products in Braşov for investigation.

Subfossil oak partially buried into the soil and timber stored in inadequate conditions (Central part of Romania- Covasna county)

Subfossil oak discovered in the Western part of the country (Timiş county)

Fig. 1. *Subfossil oak in situ or after digging in different regions of Romania*

2. Objectives

The present paper is a literature review and it aims at acquiring knowledge and understanding of subfossil wood in contrast with recent wood, by focusing on oak.

The study envisages new opportunities for the research of valuable wood resources, as well as the appropriate utilisation of this material in Romania.

3. Understanding the Material

Starting this study, we were faced with some confusion related to specific terms such as: fossil/fossilised and subfossil wood. Therefore, comprehensible definitions were required.

Fossil wood is a mega plant whose remains can be found in sedimentary rocks [12]. The fossilisation process starts when the trunks are buried and remain in anaerobic conditions. The minerals precipitate within the cellular spaces and crystalize after the saturation of wood with groundwater. The slow process of fossilisation involves the transformation of the cell wall substance into highly condensed compounds (coalification) or its substitution by minerals (silicification) [9].

Generally, the fossilization process is associated with marine or volcanic hydrothermal water [20], [30].

Subfossil wood is non-petrified wood which has been hidden **for hundreds to thousands** of years in rivers, bogs or glacial sediments [16]. This wood originates from bottomland forests. Over time, as a result of cyclical flooding, this biotope disappeared [9]. Another definition proposed by Pearson et al. [24] revealed that subfossil wood has the potential to be preserved for a long period of time, but suggested that the woody structure is not changed as in fossilization.

Subfossil tree trunks were dated from the Post-Glacial Period and were discovered into alluvial deposits of temperate and central European rivers (Vistula, Danube, Warta, Maine, Morava, Middle Rhine, and Rhône).

Oak wood is one of the most common species existing in subfossil form, but there are also: pine, spruce, fir, elm, poplar, beech, ash, and birch, depending on the geographical area and the forest habitat. Oak invaded Europe in 12,000 BP and has become a dominant woodland species in most parts of the continent. The successful process of oak colonisation was the result of its adaptation to different ecological conditions: soils, climates, altitudes, and the regenerative power from seeds. Moreover, oak has a wide range of uses for humans. It is an excellent material for construction, furniture, wooden products, barrels, it is durable and resistant.

Subfossil oak is known as "black wood" due to the colour and long-term storage and reactions of the sediments´ iron compounds to tannins present in oak [35].

Many authors explain the wood preservation under the surface for thousands of years by a high and constant ground water level, no oxygen access, low redox potential, and optimal pH [8], [14], [16-18], [22]. Under these conditions complex physical and chemical processes occur.

The chemical and morphological structure changes are reflected in the wood appearance and properties [16].

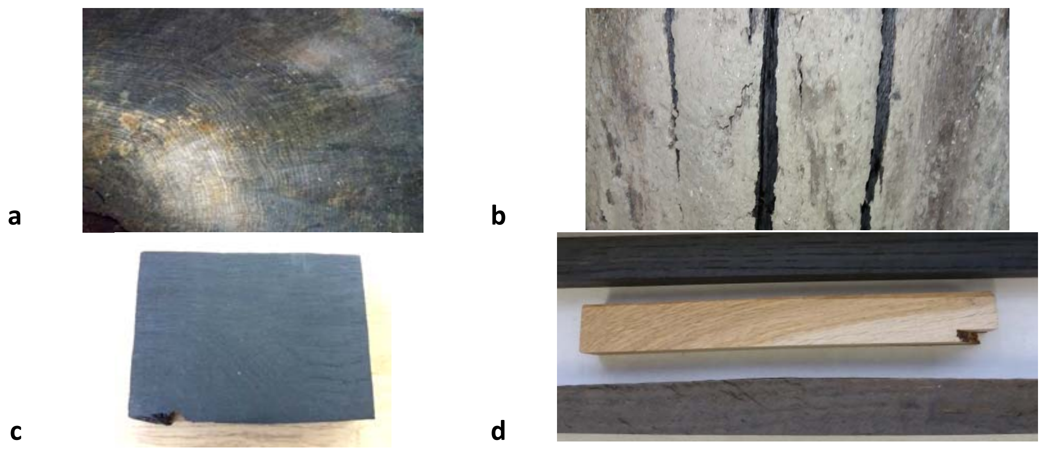

Fig. 2. *The appearance of subfossil oak wood*
(a- cross section, b- external part with sediments and cracks, c-processed wood, d- subfossil oak in different colours vs. new oak)

Figure 2 shows some macroscopic images of subfossil oak wood originating from the Romanian sites previously presented.

In the cross section the narrow and equal annual rings can be seen (Figure 2a), as well as the exterior of the trunk, covered with sediments from the riverbed (Figure 2b). The tendency of cracking is evident and this inconvenience must be considered when storing and drying, preserving and processing the material. The black colour is observed in different shades after processing, in contrast to the new wood (Figure 2c and 2d). This is an advantage when using the material in design projects. As a result, subfossil oak has become popular in the last few years.

3. Dendrochronology

The shape of the tree rings in living trees is important information about past environmental changes. Tree-ring chronology of living trees can be used to analyse radial growth variability during different periods in history. Combining these records with the tree rings from dead trees helps extend the chronologies further back in time [13].

In historical research, the most important method used for the dating of wood is dendrochronology. The samples which could not be dated by the available standard chronologies were tested using the radiocarbon method (^{14}C) [29], [31].

The standard chronology is created for each tree species by gradual overlapping of growth ring sequences towards the past [17]. The tree-ring width reflects the environmental conditions in the past. Even so, two aspects must be considered when a subfossil tree is dated:

1. The dating by standard chronology is not possible when the tested area is not sufficiently long. In this case, the radiocarbon method, based on the proportion of stable and unstable carbon is used [16, 17], [36]. The radiocarbon method allows for dating materials of up to about 50,000 years [37];
2. The nature of the soil sediments must be considered as specific components of dating. If subfossil tree trunks are *in situ*, whether they are rooted or unrooted is important. The chronology for rooted trees is clearly established as palaeosol - an ancient soil in a relatively unaltered state, formed on landscapes of the past [28]. The situation is different when the subfossil trunks are found unrooted. These trunks can be longer and a period of time has passed between their death and their final incorporation into the sedimentary soil. Tree trunks fall in the riverbed and they can be buried in situ or transported and reworked within the river channel [7].

In Romania the first dendrochronological data series were elaborated after 1985 according to the International Tree Ring Data Bank for: spruce, fir, sessile oak, and pedunculated oak [23]. Special attention was given to the dating of wooden monuments or medieval buildings from Maramures county [38].

Since 2015, RoAMS laboratory has begun a series of ^{14}C determinations on samples from archaeological sites spread all over the Romanian territory, as well as objects from the cultural heritage [29].

Obviously, oak has great significance in dendrochronological European studies. Its importance and usefulness for the study of different sites in Europe have been highlighted [24].

Table 1 summarises a dendrochronological data sheet from the studied literature, focussing on subfossil species from different locations in Europe. Only few data are presented by citing the reference. As can be seen, the results are expressed on a different time scale, specific for the archaeology and geology systems. The age of the investigated wood varied from hundreds to thousands of years. The symbols are noted in the bottom row.

Table 1

Chronology of subfossil wood (selective data from the literature)

No	Wood species	Location	Methods/Results		References
			Dendrochro-nology	Radiocarbon ^{14}C / yr.	
1	Subfossil Pine, Subfossil Oak Wood elements of trackway construction	Germany	3000 BC	-	Leuschner et al., 2007 [21]
2	Subfossil Oak	Czech Republic	900-1000 AD 1300-1600 AD	210-6170 BP 1800-3900 BP	Kolar et al. 2012 [16]
3	Subfossil Pine, Subfossil Oak (unrooted)	Poland	950-460 BC 1500-1600 BC	2360-2820 BP 3190 BP	Barniak et al., 2013 [5]
4	Subfossil Spruce and Silver fir	Maramureş Romania	255- 388 AD 985- 1023 AD	1039 – 1717 BP	Arvai et al., 2014 [1]
5	Subfossil Pine Subfossil Oak	France		10 000 BP 8000- 8400 BP 4150 -4950 BP	Carozza et al., 2014 [7]
6	Subfossil Oak (Many sample groups)	Balkan Rivers	2113-1965 BC 1588-1551 BC	4062–3914 BP 3357-3500 BP	Pearson et al., 2014 [24]
7	Subfossil Oak	Belarus	1575–1747 AD	5782–5612 BC	Vitas et al., 2014 [33]
8	Subfossil Oak	Mari el Republic / Russian Federation	550 - 200 BC 1020 - 1170 AD 1160 - 1310 AD	2300 BP 950 BP 750 BP	Barcik et al., 2015 [4]
9	Subfossil Oak Beech, Elm, Poplar	Moldova and Siret Rivers- Romania	-	3000-4000 BP 1000 BP 785 BP 650 BP	Radoane et al., 2015 [25]
10	Subfossil Scots pine	Sweden	1300-1550 AD	-	Zhang et al., 2015 [34]
11	Subfossil Oak Elm	N-E Romania	-	700-7000 BP	Kern and Popa, 2016 [14]
12	Subfossil Scots pine stem	N-E Finland	1100-1700 AD	-	Helama et al., 2020 [13]
13	Subfossil *Pinus cembra* trunks	Eastern Carpathian Romania	1009-1709 AD	102-1030 BP	Sava et al., 2019 [29]

BC – Before Christ

AD = CE – Anno Domini (Common Era), after Christ's birth.

BP – before present – Before Present". The most commonly used convention in radiocarbon dating.

4. Chemical Composition

Even if they are old trees, the subfossil tree trunks still mainly consist of lignin and cellulose [7].

Subfossil wood has a different chemical composition, in correlation with the conditions where it was deposited [16]. When comparing the chemical composition of subfossil wood with recent wood, it differs by a lower proportion of hemicelluloses.

Kolar et al. [17] explained that hemicelluloses are easily eluted in a humid environment [16]. Therefore, when water affects wood deposited in the soil, some substances are removed and carbonate of lime and silica remain in the wood surface [16, 17].

For oak wood which was over 8,000 years old, found in excavation in the `80s in the USA, both the content of lignin and cellulose increased compared to recent oak wood [9]. When comparing the chemical composition of subfossil pine of 12,500 years old with recent wood, Fejfer et al. [8] reported a 30% cellulose content in the subfossil wood compared with 50% in the recent wood. The lignin increased twice than in the recent wood (55% compared with 27%) [8].

Recent research [10] carried out on historical oak wood from Iaşi – Romania dated in the fourteenth century revealed that the amount of α-cellulose in the old wood and in recent wood was not significantly different (41.72% and 39.7%, respectively) (see Table 2). The hemicelluloses content in the old oak samples was significantly lower compared to the recent samples (19% compared with 24%). The lignin content increased: 35.2% in the old wood compared with 27.4% in the recent wood. During the aging of wood in soil contact, the hemicelluloses could be degraded. The ash content increased for the old wood (2.24% compared with 0.23% in the recent wood) (see Table 2).

Baar et al. [3] reported in their study that the lignin content in the subfossil oak wood was much higher by about 20–30% than in the recent wood. Also, the same authors concluded that when the wood was buried in the soil, the amount of wood components soluble in water and/or methanol, as well as of phenolic compounds, diminish [3]. Thus, 3000 year-old subfossil oak contained 60% less phenolic compounds in comparison with more recent subfossil oak (of 1000 years old). As expected, the percentage of mineral substances (ash) data indicated a higher concentration in subfossil oak than in recent wood (1.86% and 0.38%, respectively) [3].

Other studies on subfossil oak and recent wood [16, 17] revealed that the proportion of cellulose did not change considerably with age. They have also found 23-41% more lignin in subfossil oak than in recent wood. The greatest difference between the two types of wood is the ash content which grows from the trunk centre to its most exposed parts. The ratio of ash content in subfossil oak to recent oak reaches 5:1 in the outer zone and 16:1 in the centre, according to Kolar et al. [17]. The chemical analyses of ten elements indicated a great amount of calcium, concluding that subfossil wood has gone through a calcification process [16, 17].

5. Wood Properties

Subfossil wood is considered a valuable raw material, interesting for wood marketers and the wood industry, as it changes its properties during exposure in soil over a long period of time. Literature sources contain information on the structural, physical, and mechanical properties, and the conservation of wood dated hundreds and thousands of years ago [1-34].

Table 2

Selection of the main properties of subfossil wood vs. recent wood

Properties		OAK			References
		Subfossil	Recent	Effect	
Chemical composition [% w/w]	Cellulose	50 (*41.7*)	55.2 (*39.7*)	↓	Baar et al., 2014 [2] (*Ghavidel et al., 2020 [10]*)
	Hemicelluloses	21.1 (*19*)	22.3 (*24.4*)	↓	
	Lignin	28.9 (*35.2*)	23.5 (*27.4*)	↑	
	Mineral subst. [%]	1.86 *(2.24)*	0.38 *(0.23)*	↑	Mankovski et al., 2016 [22] (*Ghavidel et al., 2020 [10]*)
Anatomy [150 yr]	Annual ring width [mm]	1.4	2.1	↓	Mankovski et al., 2016 [22]
Physical	Density [kg/m^3 MC 12%]	846	662	↑	Mankovski et al., 2016 [22]
		735	690	↑	Kolar and Rybniček, 2010 [18]
		637			Veizovic et al., 2018 [32]
	Volumetric swelling [%]	20.5	13.2	↑	Mankovski et al., 2016 [22]
	Tg. Swelling [%]	11.23 (12.3)	8.40	↑	Kolar and Rybniček, 2010 [18]
		16.9	-		Veizovic et al., 2018 [32]
Mechanical properties	Bending [MPa]	125	121	No change	Mankovski et al., 2016 [22]
	MOE [MPa]	4408	-		Kolar and Rybniček, 2010 [18]
		9600	10 050	↓	Mankovski et al., 2016 [22]
	Compression parallel [MPa]	29.3 (38.5)	43	↓	Kolar and Rybniček, 2010 [18]
		80	72		Mankovski et al., 2016 [22]
	Hardness [MPa]	41.6	52.4	↓	Kolar and Rybniček, 2010 [18]

Legend: ↓- increasing, ↑- decreasing, - no values

5.1. Anatomical Structure

The macroscopic features related to the annual rings (width and latewood proportion) of subfossil pine indicated similar values in recent pine [8]. Considering the age of the pine tested (dating from 12,500 BP), the wood was quite well preserved.

Research on subfossil and recent oak revealed that ring width does not differ. The mean surface of all the earlywood vessel area increases for both subfossil and recent oak samples. Generally, the results of the anatomical parameters analysis do not differ when comparing subfossil oak with recent oak [16, 17].

The microscopic analysis of subfossil wood evidenced no general deformation of anatomical elements. The microstructure is distinctive and the identification of the wood species is not so difficult [26]. Unfortunately, the surface was more brittle, which indicates a cell wall degradation, most likely bacteria, fungi, acid and alkali [10]. Some inorganic deposition into the cell lumen was identified. Moreover, the microscopic investigation allows the assessment of wood decay to the cell level and it is a useful instrument in combination with cross-dating of wood [27].

5.2. Physical Properties

Wood density is a key property which defines wood quality and use. It has great variability according to: species, growth condition, and tree development stage. Also, it is closely related to moisture content. Density (specific gravity) values are usually based on oven dry weight and volume at 12% moisture content.

Studies on subfossil oak reported an increasing wood density of about 850 kg/m^3 over the average of typical contemporary wood (approx. 670 kg/m^3), due to the mineral compounds including iron compounds that saturate the wood [22]. When discussing pine, a significantly lower density was recorded but the density varied with the tested area from the trunks. Accordingly, for subfossil pine the value was 270 kg/m^3, compared with 483 kg/m^3 in recent wood [8].

Generally, researchers agreed that a high variability of density was reported along the stem radius and its value depends on the location, the conditions of deposition, and also the degree of degradation [4], [11], [16], 34].

Density influences the water transport into the wood, being interconnected with dimensional stability.

Some authors concluded that subfossil oak wood has a tendency to swell and shrink twice as much as recent wood [18, 22]. The average volumetric swelling of subfossil oak wood was 20.5%, whereas in contemporary oak wood, with a larger tree-ring width, the volumetric swelling was only 13.2% [22].

The increase in wood swelling was not so evident when comparing subfossil and recent pine. In tangential direction, the swelling of subfossil pine was 7.5-8% compared with 6.2% for recent pine [8].

Nevertheless, this property must be correlated with wood species, age, origin of material, anatomy, density, and cracks in the wood.

5.3. Mechanical Properties

Generally, the mechanical properties of subfossil oak wood are inferior to the properties of recent oak [16, 22]. Veizovic concluded that the mechanical properties of subfossil oak are lower by 10-40% than those of recent oak [32].

Compression strength parallel to the grain of subfossil oak corresponds to about 70–80%, in some cases even 50%, of the strength of recent oak. The studied literature indicates lower values of subfossil oak hardness even though the cell structure was modified by harder minerals [18].

For other subfossil species such as elm, the specific underground depositions did not significantly change the microstructure and bending strength of the wood [26].

5.4. Durability

Some durability tests showed that subfossil oak heartwood is more susceptible to decay than samples of heartwood from recent oak [3]. The age of the subfossil oak itself had no influence on its durability. The mass loss for subfossil oak was 2–3 times lower than for the recent wood sample. It ranged between 5.0% and 11.1% depending on the fungus species. The lower durability is assigned to a lower amount of phenolic compounds with fungicide effect [3]. Due to the chemical changes and their effects on durability, subfossil oak is not a material recommended for exterior use [3].

Table 2 presents the studied properties and experimental data (where present) obtained for subfossil oak compared with recent wood. The subfossil oak was dated according to different studies and authors (see reference table) 1000 BC or varies 2490-2190 BC but some subfossil wood was only 150 years old.

5. Conclusions

Subfossil wood can provide opportunities for insight into history, climatic changes, environmental reconstruction in different world regions, tree evolution, and wood research.

The study is opportune and useful in the context that subfossil wood is not well known and used in Romania and a database comes to fill the gap of information in this field.

Also, the study investigates some of the main properties of subfossil wood required for research or practical uses, while also identifying the limitations of the material.

Moreover, a multidisciplinary approach between geologists, foresters, engineers, and designers could be achieved in the future, taking into account subfossil wood.

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