#### WOOD RESEARCH

63 (6): 2018 959-970

# TESTING OF WOOD PHYSICAL PROPERTIES IN OAK SPECIES (QUERCUS ROBUR L., Q. PETRAEA (MATTS) LIEBL. AND Q. PYRENAICA WILLD.) FOR COOPERAGE. PART II: WOOD GRAIN

Diaz-Maroto I.J.
Universidad De Santiago De Compostela, Escuela Politécnica Superior
De Ingeniería
Lugo, España

Tahir S. École Superieure Du Bois Nantes, France

(RECEIVED AUGUST 2018)

#### **ABSTRACT**

Our research about wood physical properties of *Quercus robur* L., *Q. petraea* (Matts) Liebl., and *Q. pyrenaica* Willd., for cooperage was founded on the calculate of wood grain to estimate the change of this property in the Galician oaks. Overall, 45 trees were selected in 15 oak forests of the provinces of Lugo and Ourense (Galicia, NW Spain), of which we obtained 45 thin slices of wood at 60 cm tall on the trunk, and 194 wood samples parallelepipeds of  $2 \times 2 \times 4$  cm  $\pm$  1mm.

The grain of *Quercus pyrenaica* and *Q. robur* are similar to oaks Limousin region, France. *Quercus petraea* is comparable to the Vosges oaks. Now, both regions are among the most important sources in quality oaks for manufacturing barrels. Our global objective was to realize a detailed description on the physical properties of wood of these species for its possible use in industry cooperage. For this, the aim of this second work was to continue with the study of wood grain.

KEYWORDS: NW Spain, Galician oaks, cooperage use, physical properties.

#### INTRODUCTION

Forest management has a significant impact on the sustainability of natural forest ecosystems, and oak forests in particular, by applying traditional forestry treatments (Bouchon

and Trencia 1990). Forestry practices were developed and updated in those European countries where thousands of hectares of oaks are managed for centuries (Bary-Lenger and Nebout 1993). The state in our study area is different, since there is less knowledge about management to be applied to these forests. As a result of the different use and conservation status, current Galician oaks have a full range of ages and qualities (Diaz-Maroto et al. 2005). Coppice forest dominates and it requires persistent management otherwise the stands will age and become stagnant (Diaz-Maroto et al. 2006a, 2010). Galician native forests were exploited intensively applying forestry practices inadequate (i.e., pollarding and felling of the best trees). Over the past century, as a result of rural depopulation and social requirements, there was a radical change from overuse to total lack of exploitation (Diaz-Maroto and Vila-Lameiro 2007). Now, as indicated by Directive 92/43/EC, are habitats of Community interest, being basic in many areas to implement a sustainable development.

Oak forests live in an area of 246,445 ha in Galicia, 18% of the total forestry area (MAGRAMA 2011). Forest management is practically not there and the use of its wood is limited for firewood. The Galician oaks are scary used in the industry, since the presence of the numerous small plots and a large number of owners don't facilitate its use (Diaz-Maroto et al. 2005). The global objective of our research was to study the anatomical and physical features of wood of Galician oaks for the manufacture of wine barrels, and to do a description on their physical properties for its use in the cooperage as key for sustainable rural development. For that, the aim of this work was to determine the wood grain to understand their variation in the Galician oaks.

#### **MATERIAL AND METHODS**

#### Research area

The research area is located in the Autonomous Community of Galicia. The middle altitude is 508 m and slopes of more than 20% present in half of the land. The lithology is diverse, even though there is a dominance of siliceous substrates; the climate is varied, but generally classified as Humid Oceanic with a Mediterranean influence in some zones. Annual precipitation varies between 600 to more than 3000 mm and the average temperature is near to 13°C (Diaz-Maroto et al., 2006a).

#### Sampling design

The research area was considered one unit where the zones for sampling were selected including an enough number of oak stands on the basis of the Forest Map of Spain. The minimum area ranged between 0.5 and 1 ha, which avoided problems of the edge effect. Finally, 45 trees were chosen on 15 stands of the eastern provinces of Galicia, Lugo and Ourense, i.e., we have cut 45 thin wood slices at 60 cm tall on the trunk and obtained 194 samples with a parallelepiped shape and dimensions of  $2\times2\times4$  cm  $\pm$  1mm.

As 69% of Galicia is above 600 m of altitude, a lot of the stands are located on land with steep slopes. This fact is a problem because a strong slope encourages the creation of the tension wood with a heart off centre, not appropriate to manufacture barrels for wine aging.

Tab. 1 show the characteristics –quality– of the wood slices regarding to growth rings from trees felled (from normal tree to heart strongly off centre) (Vila-Lameiro and Diaz-Maroto 2005).

Characteristics	Quercus robur	Quercus petraea	Quercus pyrenaica	Total
Normal tree	7	6	5	18
Heart a little off centre	4		2	6
Heart off centre	4	8	3	15
Heart strongly off centre	2	1	3	6
Total	17	15	13	45

Tab. 1: Characteristics -quality- of the slices of wood from trees felled.

#### Statistical analysis: Image J

ImageJ software is a processing program public domain images with which we can calculate, among other features, the area of the different parts of the tree trunk, i.e. bark, sapwood and heartwood, as well as their proportion. Its high precision allows using a wide wooden sample, in addition to considering the uniqueness of each sample. ImageJ was designed according to an open architecture than supplies extensibility via Java plugins. It is multithreaded, so time-consuming processes such as image file analysis can be done together with other. It can estimate area and pixel value statistics of user-defined options. Also can calculate distances and angles and create density histograms. It supports standard image processing functions, e.g. contrast manipulation, sharpening, smoothing, edge detection and median filtering. Spatial calibration is available to provide real world measurements in millimetres (Ferreira and Rasband 2012).

#### Wood density

Wood density fluctuates mostly with tree species, growth conditions and the tree part. The stem generally has a higher density that the branches, while fast growth is related to low wood density. The knowledge about the "density of wood" is useful for estimating the porosity (Pot et al. 2013). Bakour (2003) defines it as the relation of the wood mass to the volume it occupies, in saturated state:

$$\rho = M_0 / V_s$$

where: ρ - density in g·cm<sup>-3</sup> or kg·m<sup>-3</sup> units (Gelhaye and Guilley 2000),

M0 - oven-dry mass of the wood,

Vs - saturated volume.

In the various methods for determining the density, the calculation of the oven-dry mass  $(M_0)$  is always done in the same way. Oven-dry weight is measured from a wood sample by drying it between 24 to 72 hours at 103°C a ventilated oven until constant weight. The samples should be weighed quickly with a precision balance one by one immediately after being taken out of the drying oven (Pasztory et al. 2014).

There are different aspects we need to be aware to comprehend the diverse methods to get the saturated volume  $(V_s)$ : i) it is basic to be sure all the empty spaces of the wood are filled with water; ii) restrict the dispersion in water of soluble substances because they could represent up to 4% of the volume of the sample; iii) differences in density and porosity may arise from differences in the anatomy of the wood modified by the extractives (Ghazil 2010). The first method requires of realizing the water immersion under a lower pressure in order to ensure a full filling of the all spaces. The second is called "The Maximum Moisture Content's method" and consists of boiling the wood samples during 15 hours (Vermaas 1988). Finally, the third implies an immersion in cold water during 24 hours minimum. Therefore, we cannot ensure a smaller dispersion of soluble substances which does not exist in the first and second method. But, if the cold water immersion

is performed under a lower pressure, both of the difficulties are overlooking. For these reasons, we will use this method.

#### RESULTS AND DISCUSSION

#### Scheme of the method

Definition of the scale

First, we have taken photographs of the samples using a Canon EOS 550D which uses a special structure to ensure optimal light and the distance between the camera and the samples is always the same. The first step is to define the spatial scale of the image so measurements can be obtained in calibrated units, in our case, centimetres. Before using the scale command, we need to make a line selection corresponds to a known distance. ImageJ will have automatically filled in the distance in pixels based on the length of the line selection (Ferreira and Rasband 2012).

#### Selection of the polygons

Subsequently, the second step is to use the polygon selection tool to create polygons irregularly shaped, defined by a set of linear segments. To generate a polygon it is necessary to click repeatedly with the mouse to select linear segments corresponding to the border of different areas, although ImageJ has an automatic selection tool—wand tool—. The determination of the bark is the most difficult, because its perimeter is irregular, and sometimes it's complex to distinguish the correct limit (Vila-Lameiro and Diaz-Maroto 2005).

#### Results and interpretation of the wood density

The selection program of the samples follows the criteria following:

- 1. Three different species of oak for use in cooperage from the same geographical region: Quercus robur, Q. petraea, and Q. pyrenaica
- 2. Three distinct types of wood: sapwood, heartwood, and juvenile wood
- 3. Three types of tree regarding to growth rings: with rotten heart, with an off-centre heart, and tree with a normal heart

Tab. 2 shows the sample division according to the wood type and species.

Tab. 2: Distribution of the sample by wood type and species.

Wood type/Species	Sapwood	Heartwood	Juvenile wood	Total
Quercus robur	33	27	8	68
Quercus petraea	19	27	6	52
Quercus pyrenaica	29	34	11	74
Total	81	88	25	194

Tab. 3 presents the distribution of the sample by tree type and species. Only the thin slices from *Quercus petraea* present a rotten heart which is a peculiarity of the area where these trees were felled.

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Tree type/species	Tree with rotten heart	Tree with an off-centre heart	Normal tree	Total
Quercus robur	0	19	49	68
Quercus petraea	17	0	35	52
Quercus pyrenaica	0	16	58	74
Total	17	35	142	194

Tab. 3: Distribution of the sample by tree type and species.

Normally, as we mentioned above, three trees were felled by plot. The notable slopes motivate the existence of off-centre hearts. This wood type is not suitable for the production of barrels (Pot et al. 2013). This is the reason why sometimes there are only one or two trees without off-centre heart per plot for the measurement of wood density. In each individual tree, different samples were obtained of sapwood and heartwood in function of its proportion.

Wood density of 142 samples from 18 normal oak trees give us a value mean equal to 648 kg·m<sup>-3</sup> with a coefficient of variation (CV) of 7%, and extreme values ranging from 498-906 kg·m<sup>-3</sup> (Tab. 4). Gelhaye and Guilley (2000) found an average of 553 kg·m<sup>-3</sup> over a set of 90 samples of *Quercus robur* and *Q. petraea* in the forest of Little Charnie (France), and Bakour (2003) affirms to have obtained a similar average, 530 kg·m<sup>-3</sup>, with a sample of 588 test tubes in French forests.

In contrast, Deret-Varcin (1983) got a lower value of wood density, 450 kg·m<sup>-3</sup>, on a set of 140 samples of the same species in the forest of Morimond, France. However, the data of Vivas (2000) in a *Quercus robur* stand of the northern Portugal were higher, 650 kg·m<sup>-3</sup>, and like to ours, because the method of measurement was the same and the site is similar (Lebourgeois et al. 2004). Reading these studies allows us to observe the variability of the oak wood density according to the geographical origin and maybe the measurement method.

#### Wood density variability according to the geographical origin

Many variables have an impact on what we know as "geographical origin". In our research on sessile oak (Diaz-Maroto et al. 2006b) cited more than different sixty parameters: climatic, soil and physiographic.

Now, our aim will be study the influence of geographical origin, on the overall, in the wood density of the Galician oaks. To properly understand the results, the data were classified by species and wood type because discrimination between sapwood and heartwood involves differences among 12 and 20% should not be attributed only to the geographical factor (Alanon et al. 2011a). It is difficult to identify a tendency because of the small number of measures. However, in Tab. 4 is shown as for similar growth ring size and age, the wood density varies significantly: e.g. there is up a difference to 132 kg·m<sup>-3</sup> in *Quercus pyrenaica* samples.

Only samples of *Quercus petraea* have values extremely close. This fact could be explained by the geographical proximity of the plots as well as the similar climatic and physiographic conditions (Alanon et al. 2011b). Also, it is interesting to observe the values of the coefficient of variation within the same plot (Tab. 5); in general, the wood density values show a low variation at the plot level.

			Growth		Wood density (kg·m <sup>-3</sup> )				
Species	Wood type	Wood samples	ring size (mm)	Age	$\bar{x} \pm \sigma$	Minimum	Maximum	Coefficient variation (%)	
	Sapwood	25	5.94	45	584 ± 55	498	678	9	
Quercus	Heartwood	26	5.82	45	716 ± 61	584	825	9	
pyrenaica	Juvenile wood	7	6.13	45	732 ± 47	673	811	6	
Total/Wei	ghted average	58	5.91	45	661			8	
	Sapwood	27	7.38	43	610 ± 72	486	744	12	
Quercus	Heartwood	17	5.97	49	695 ± 68	622	906	10	
robur	Juvenile wood	5	5.47	51	677± 27	626	703	4	
Total/Wei	ghted average	49	6.70	46	646			8	
	Sapwood	15	3.98	67	583 ± 31	532	628	5	
Quercus petraea	Heartwood	15	4.00	67	671 ± 15	650	712	2	
	Juvenile wood	5	4.08	64	640 ± 16	608	652	3	
Total/Weighted average		35	4.00	67	629			3	
Global total		142			648			7	

Tab. 4: Wood density, growth ring size and age by species and wood type.

## Other factors affecting the variability of oak wood density: species, growth ring size, age and wood type

Quercus pyrenaica has the highest wood density with an average of 661 kg·m<sup>-3</sup>, followed by Q. robur, 646 kg·m<sup>-3</sup>, and Q. petraea, 629 kg·m<sup>-3</sup> (Tab. 4) which is in line with Fernandez-Parajes et al. (2005). However, these data disagree with those obtained by other authors (Deret-Varcin, 1983, Bakour, 2003), especially Quercus petraea, where the resulting values were higher. The wood samples of this species come from a mountain area with an altitude greater than 1200 m and steep slopes. The environment –growth– conditions are complex and affect the width of rings: only 4 mm of average, the lowest average of growth ring size.

A lower ring width in oak species results in a lower texture (Vila-Lameiro and Diaz-Maroto 2005); the percentage of early wood is more significant than the late wood (Pasztory et al. 2014). The oak wood has a first porous area since it has many vessels in the early wood (Bary-Lenger and Nebout 1993). So a lower texture involves more vessels on the slice surface, resulting in a lower wood density (Pot et al. 2013). Also, the higher average ages of *Quercus petraea* as well an influence on density.

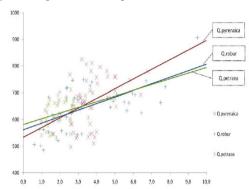
Fig. 1 shows a positive correlation between the growth ring size and the wood density. A high density agrees with a maximum of heartwood and a small proportion of vessels. In the oaks, this small proportion of vessels results in an important proportion of late wood –i.e. a strong texture– (Gelhaye and Guilley, 2000, Lehringer et al. 2008).

The slope model for *Quercus pyrenaica* is more significant than for *Q. robur* and *Q. petraea* due to the higher density observed in this species. (Fig. 1). The low value of the Pearson coefficient of *Quercus petraea* is due to the lack of data, only 35 against 58 for *Quercus pyrenaica* (Tab. 4), associated with a large range of ages.

Regarding to the density variability in function of the age, we don't even need to note at the Pearson coefficient to find this model does not work. Bakour (2003) showed a negative correlation between age and density for *Quercus robur*. Modelling on 123 trees makes it possible to obtain the equation following:

Density = 
$$600.498 - 0.564 \times age$$
;  $R^2 = 0.262$ 

The modelling slope is only -0.564. However, the slope of the function between the growth ring size and density is 24.482 for *Quercus robur* (Fig. 1). Therefore, we can deduce the influence of age on wood density in this species is less important that the influence of width of growth ring.



Quercus robur: y = 24.482 x + 561.79;  $R^2 = 0.3972$ 

Quercus petraea: y = 21.219 x + 581.99;  $R^2 = 0.1844$ 

Quercus pyrenaica: y = 36.296 x + 533.39;  $R^2 = 0.2116$ 

Fig. 1: Relationship between density (kg m<sup>-3</sup>) and growth ring size (mm).

Finally, concerning to the wood type, as shown in Tabs. 4 and 5, whatever the species, the sapwood has the lowest density with values between 12 and 20% lower than average density of heartwood and juvenile wood (Deret-Varcin 1983, Lehringer et al. 2008). Also, it is interesting to note the sapwood has the highest CV (Tabs. 4, 5); it is an area with moderately variable density (Pasztory et al. 2014).

From an anatomical viewpoint, the heartwood only contains dead cells because it has undergone chemical and sometimes physical transformations (Hacke and Sperry, 2001). The ways of sap flow can be closed (closing bordered pits of conifer tracheids) or become blocked (blocking of vessels broadleaved trees by membrane extensions called "tyloses") (Ghazil 2010). Therefore, we can assume that these obstructions leave less space for air and water, which explains a higher density than in the sapwood. Sapwood consists of living cells, the non obstruction of the vessels are critical to the flow of sap. It is complicated to conclude on the density of the juvenile wood. For the species *Quercus robur* and *Q. petraea*, they have a density slightly lower than heartwood. For *Quercus pyrenaica*, wood density is slightly higher than in the other species (Tab. 4).

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Species	Wood type	Province	Trees	Growth ring size (mm)	Age	Wood density (kg·m <sup>-3</sup> )	
	vvood type		(samples)			$\overline{x} \pm \sigma$	CV (%)
		Ourense	1 (3)	1.27	50	609 ± 22	4
		Ourense	2 (8)	3.26	46	548 ± 16	3
	Sapwood	Ourense	1 (2)	1.60	48	547 ± 1	0
		Ourense	1 (4)	3.26	44	523 ± 22	4
Quercus		Ourense	2 (8)	3.42	37	651 ± 26	4
pyrenaica		Ourense	1 (2)	2.55	50	768 ± 2	0
	Heartwood	Ourense	2 (8)	3.86	46	661 ± 48	7
		Ourense	1 (4)	3.45	48	674 ± 27	4
		Ourense	1 (4)	4.48	44	736 ± 23	3
		Ourense	2 (8)	4.09	37	729 ± 25	3
	Sapwood	Ourense	2 (15)	4.62	29	637 ± 87	14
		Lugo	2 (8)	2.13	59	568 ± 30	5
Quercus		Lugo	1 (4)	1.63	57	598 ± 25	4
robur		Ourense	2 (5)	4.51	29	688 ± 123	18
	Heartwood	Lugo	2 (8)	2.71	59	676 ± 30	4
		Lugo	1 (4)	3.35	57	740 ± 10	1
Quercus	Samura J	Lugo	3 (7)	1.59	80	568 ± 33	6
	Sapwood	Lugo	3 (8)	1.73	53	596 ± 28	5
		-	- (-)				_

Tab. 5: Wood density, growth ring size and age according to the geographical origin.

### 3 (8) The properties of Galician oaks for the manufacture of barrels: wood grain

A lot of parameters affect exchange between wine and wood. The species, the wood porosity, the grain, the aging method, the toasting type, the volume of the barrels and the industrial process offer a many possibilities for the aging of quality wines.

2.34

2.66

80

53

 $674 \pm 17$ 

 $667 \pm 13$ 

#### The importance of wood grain

Heartwood

The aging in oak barrels is an essential step in the obtaining of quality wines. During this process, two important phenomena occur: i) the oxidation of some substances of the wine due to the infiltration of oxygen; ii) the distribution the aromas and tannins of oak wood. Feuillat et al. (1994) define the barrel as an "active interface" between a liquid (wine) and a gaseous environment (air of the wine cellar) that determines the exchange phenomena between the two environments. Exchanges occur by the joints of the staves, and also through of the staves themselves (Prida 2006).

In addition, the wood fibers must be straight and not cut to be watertight. The wood with nodes is also unsuitable for the same reason. Coopers use the concept of grain. This is a test makes it is possible a fast and reliable visual classification. The criteria most commonly used are the geographical origin of wood and their grain, that is to say, the width of growth rings and the regularity. In France, they usually use the following classification (Vivas 1995):

Crude-grain: width of growth rings to 5-4 mm.

Lugo

Lugo

- Medium-grain: width of growth rings to 4-2 mm.
- Tight grain: width of growth rings to 2-1 mm.
- Very tight grain: width of growth rings less than 1 mm.

Tab. 6 shows the results obtained by Vivas (1995) in different regions of France compared with the results obtained in our research on the wood of different Galician oaks.

Data		Geographical origin	Number of trees	Growth	Number of growth rings by cm			
source	Species			ring size (mm)	$\overline{x}$	Heterogeneity (min/max)	CV (%)	
Vivas (1995)	Quercus robur	Limousin			2.22	0.42	28	
	Quercus petraea	Vosges	180		3.44	0.42	29	
	Q. pyrenaica	Centre			5.56	0.65	14	
Galician oaks wood	Quercus robur	Galicia	5	3.5	1.77	0.36	40	
	Quercus petraea	Galicia	4	2.2	2.57	0.63	20	
	O. pvrenaica	Galicia	7	3.5	1.74	0.57	19	

Tab. 6: Comparison of oak wood grain between some regions of France and Galicia.

With values of growth ring between 2.2 and 3.5 mm, the Galician oaks wood has a medium grain (Vivas 1995). The number of growth rings per centimetre indicates a greater growth than in the regions of Vosges and the Centre of France (Tab. 6). Finally, it notes despite a smaller number of trees used in our study, measures of heterogeneity and dispersion (characterized by the CV) is relatively similar.

#### CONCLUSIONS

Because the amount of data used in our work have not sufficient, has not been possible to obtain a precise correlation between the variability of wood density and tree age. However, if we have obtained adequate results for the remaining variables related to that wood characteristic:

- On average, Galicia oak species have a density greater than of other countries with which we have made the comparison of their wood.
- With a growth ring size and age similar, *Quercus pyrenaica* has a higher wood density than *Q. robur* and *Q. petraea*. Also, density increases with ring width.
- The heartwood has a density between 12-20% higher than sapwood, there is a large density variation by geographical origin, and the density of the trees on the plot varies slightly.
- Galician oaks wood has a medium grain. The number of growth rings per centimetre indicates a greater growth than in the regions of Vosges and the Centre of France.

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\*Diaz-Maroto I.J.
Universidad De Santiago De Compostela
Escuela Politécnica Superior De Ingeniería
E-27002 Lugo
España.

\*Corresponding author: ignacio.diazmaroto@usc.es

Tahir S. École Superieure Du Bois Rue Christian Pauc F-44036 Nantes Cedex 3 France