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RELATION BETWEEN MECHANICAL PROPERTIES OF CORK FROM QUERCUS SUBER

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ABSTRACT

Cork is known as the material used for the production of wine stoppers. The specific properties of cork, e.g. low density, very low permeability to water, elastic properties and inertness have made it the best sealant for quality wine. Here we studied the relation between compression, tensile and bending stress in cork and the influence of structural characteristics of cork on its mechanical behaviour. The material was sampled from raw cork planks of good quality (class 1) and poor quality (class 4) collected at one industrial mill after post-harvest six-month air stabilization, water boiling and air drying as usually applied in cork industrial processing. The samples had densities ranging 0.123 - 0.203 g.cm⁻³ and porosities between 0.5 and 22.0%.

There are differences between the type of stress and the corresponding direction of stress. For the same direction of stress, the Young modulus in tension is higher then in bending and it is lowest in compression. The bending Young modulii were well correlated with the tensile Young modulii, because while in bending the sample is submitted to both tensile and compression stresses, the fracture occurs in the tensile zone. There were no significant differences in the mechanical properties of cork samples obtained from cork planks of different quality classes but the density is an important factor and samples with higher density showed overall larger resistance. Mechanical properties were influenced by the structural features related to the lenticular channels, namely the presence of thick walled and lignified cells that may border the lenticular channels. Keywords: Cork, mechanical properties, Young modulus, quality, lenticular channels, density,

porosity.

Notation list

Notation for the tensile tests:

Ab – load according to axial direction in a specimen from the inner part plank;

Ai – load according to axial direction in a specimen from the mid point;

Ac – load according to axial direction in a specimen from the outer part plank;

Tb – load according to tangential direction in a specimen from the inner part plank;

Ti – load according to tangential direction in a specimen from the mid point;

Tc – load according to tangential direction in a specimen from the outer part plank.

Notation for the bending tests:

Rct – load according to radial direction, where the force is applied in the other part plank and the direction of tensile/compression is tangential;

Rbt – load according to radial direction, where the force is applied in the inner part plank and the direction of tensile/compression is tangential;

At – load according to axial direction, perpendicular to the lenticular channel where the direction of tensile/compression is tangential;

Rca – load according to radial direction, where the force is applied in the other part plank and the direction of tensile/compression is axial;

Rba – load according to radial direction, where the force is applied in the inner part plank and the direction of tensile/compression is axial;

Ta – load according to tangential direction, perpendicular to the lenticular channel where the direction of tensile/compression is axial.

INTRODUCTION

The cork that is world wide known as the sealant in wine bottles is derived from the outer bark of one evergreen European oak, the cork oak (*Quercus suber* L.). Cork is a cellular material with structural and chemical features that impart it with peculiar and interesting physical and mechanical properties (Fortes *et al.*, 2004). Cork is a highly ordered structure of small, hollow and non-communicating cells (Pereira *et al.*, 1987), with suberin as the main structural component of cell walls (Pereira, 1988).

The porosity of cork given by th presence of lenticular channels is the main quality parameter and it is used to grade the cork raw material in quality classes. A good cork will have few and small diameter pores, while a poor quality cork will have lenticular channels with a large cross-sectional area. The appreciation is visual and a broad range of porosity is found in each commercial class, especially in the intermediate quality classes (Anjos *et al.* 1997; Pereira *et al.* 1996).

Some authors have described the compression behaviour of good quality cork (Rosa and Fortes 1988; Gibson et al. 1981). The Young's modulus for radial compression is roughly one and a half times that along the other two directions (Rosa and Fortes 1991; Fortes and Nogueira 1989). The compressive

properties of cork were found to vary with the density (Gibson and Ashby 1987) and cellular dimensions (Pereira *et al.* 1992). Rosa and Fortes (1991) observed that the Young's moduli in tension was higher than the observed in compression. In cork, density varies with the geometry of the cells, the undulation of cell walls and the presence of lenticular channels or other discontinuities (Rosa 1988).

The mechanical properties of cork may constitute a potential for this material to be used in innovative applications related to diverse fields. The objective of this work is to evaluate the relation between mechanical properties in cork namely tensile, compression and tree point bending tests.

MATERIAL AND METHODS

The compression and tensile properties of cork were studied on samples obtained from good quality (class1) and poor quality (class 4) cork planks and correlated with the density and porosity. The test specimens were cut from each cork plank as plates with 30mm x 5mm x 60mm edge and equilibrated in the laboratorial environment to 9% mean moisture content. Samples were taken from the cork planks in three radial positions: The inner part plank (the belly side), the outer part (the back side) and a mid position. The specimens were weighed and porosity and density calculated.

The porosity (determined by image analysis) was reported as a coefficient of porosity, in %, representing the area of pores divided by the total area, and calculated as the mean of the two faces measured in each sample. For the compression tests we measured the porosity on the perpendicular face of the load direction, for the tensile tests we measured the porosity in the higher faces parallel to the load direction and for the bending tests we measured the porosity in the higher faces perpendicular to the load direction.

The different mechanical tests were made at a constant crosshead speed of 2 mm min⁻¹ (equivalent to a strain rate of $2 \times 10^{-3} \text{ s}^{-1}$) up to a strain of 80%, for compression test, and up to fracture on the bending tests (equivalent to a strain rate of $1.4 \times 10^{-3} \text{ s}^{-1}$). The tensile test used a crosshead speed of 5 mm min⁻¹, corresponding to a strain rate of $1.7 \times 10^{-3} \text{ s}^{-1}$. Young's modulus was calculated from the average slope of the stress-strain curve between the loads of 10 N and 100 N, corresponding to strains between approximately 1% and 2.5%.

The samples obtained from cork planks of different commercial quality classes, had densities ranging 0.123 - 0.203 g.cm⁻³ and porosities between 0.5 and 22.0%.

RESULTS AND DISCUSSION

The stress – strain curves up to a strain of 80% of cork samples in compression parallel to each of the three main directions and for the two quality grades are represented in Figure 1 (Anjos *et al.* 2006). The stress-strain behaviour of cork sample in compression, for both quality classes was similar. The most significant differences referred to somewhat higher stress values for the region of larger deformations for class 4, corresponding to strains above 50%.



Fig. 1 - Stress-strain curves for the compression of cork specimens obtained from cork planks of class 1 and class 4 in the three directions (radial, axial and tangential).

The mechanical behaviour observed is common to previous results by other authors (Rosa *et al.* 1990; Rosa and Fortes 1988; Gibson *et al.* 1981; Anjos *et al.* 2005). The curves followed the pattern of an elastic region up to strains of approximately 5%, corresponding to the elastic bending of the cell walls, followed by a large plateau for strains between about 5% to 60% caused by the progressive buckling of the cell walls, with a subsequent steep increase of stress for higher strains with the crushing and collapse of the cells.

The average Young's moduli for the two quality classes observed (Table 1) are 18.3 and 16.9 MPa for the radial and axial directions, and 12.3 MPa for the tangential direction. A similar difference in Young's moduli was reported for raw cork (Rosa and Pereira 1994; Pereira *et al.* 1992), although in other

studies a lower value was found for the radial compression (Rosa *et al.* 1990). When comparing the Young's modulus, and the stress values for strains of 5%, 25%, 50%, 75% and 80% in compression for both quality classes (Table 1), it can seen that in all cases the values are high for class 1, but without significant differences of the mean values of the two quality classes (Scheffé test). The analysis of variance of the Young's moduli showed that the quality class was not a significant factor of variation or its interaction with the direction of compression; the direction of compression was a highly significant factor accounting for 60% of the total variation.

In the present study, the densities of the cork samples under compression ranged 0.121 to 0.197g.cm⁻³ (Table A.1). Anjos *et al.* (2006) observed an increasing E with density, especially for compression in the radial direction. The effect was less marked for compression in the tangential direction.

Table 1 - Compression properties of cork specimens obtained from cork planks of different commercial quality (class 1 and class 4, respectively good and poor) in the three directions (radial, axial and tangential). Mean of twelve samples and standard deviation.

Compression		Class 1			Class 4	
properties *	Radial	Axial	Tangential	Radial	Axial	Tangential
E (MPa)	17.94±2.865	16.60±1.790	13.42±1.423	18.65±3.312	17.08±2.266	11.20±1.727
σ5 (MPa)	0.61±0.057	0.59 ± 0.061	0.56 ± 0.044	0.59 ± 0.068	0.57 ± 0.104	0.44 ± 0.048
σ25 (MPa)	0.91±0.122	1.02 ± 0.142	0.89 ± 0.099	1.15±0.199	0.88 ± 0.203	0.91±0.072
σ50 (MPa)	1.28 ± 0.251	1.65 ± 0.340	1.21±0.144	1.81±0.370	1.47 ± 0.520	1.25 ± 0.488
σ75 (MPa)	5.34±0.105	5.31±0.163	5.37±0.147	6.37±2.352	6.97 ± 2.665	6.38±0.389
σ80 (MPa)	17.99±0.217	10.58±0.330	10.70±0.303	10.25±0.271	10.39±0.331	10.40±2.294

Young's modulus (E), stress for strains of 5% (σ 5), 25% (σ 25), 50% (σ 50), 75% (σ 75) and 80% (σ 80).

Adjusted stress strain curves obtained in tensile tests are shown in Fig. 2. The curves are similar for the three planes, but the inner part was slightly more resistant than the outer part of the cork plank. For all tests the cork from class 1 was statistically (ANOVA test) higher resistant under tension than that observed for the class 4.

Table 2 indicates the Young's moduli, stress and strain at fracture. The cork samples presented higher resistance in tensile that in compression. That behaviour could be explained by the structure of cork, where the stiffness of undulated plates (the cell walls) increases as the amplitude of the undulations decreases: while compression increases the amplitude, tension decreases it (Rosa, 1991). The differences observed between samples could be explained by the presence of cork defects. In the inner

part of the plank the higher tensile resistance occurred because the cells are well arranged and there are few defects, with only a few schlerenchimatic cells, and a lower porosity (Table A2).



Fig. 2 - Stress-strain curves for the tensile testes of cork specimens obtained from cork planks of class 1and class 4 in the directions axial and tangential for tree planes (inner, outer and mid)

The cork has higher resistance under tensile tests for the axial direction because there is higher residual tension on cork in tangential direction due the cork growth in the tree stem. Also the pores are not circular in the tangential section, and they have an approximate elliptical form with the higher axis in the axial direction.

The densities of the cork samples under tensile ranged 0.148 to 0.178g.cm⁻³ (Table A.2), and there is a good correlation between Young's moduli and density. The same results were observed by Gibson and Ashby (1987) and Anjos (2005).

The stress strain curves obtained in bending tests are represented in Fig. 3. The curves are similar for the different direction and similar to those obtained for the tensile tests. As in tensile, the

material resistance was lower in the tangential direction. For most tests the resistance was higher for the class 1 corks than for class 4 corks, due to the differences in cell structure and defects as explained before. The ANOVA results show that the more important factor to explain the variability was the internal tension in the specimen. The density in that tests specimen was very similar (Table A.3) and the porosity in the fracture zone was very important for the higher or lower cork resistance.

Table 2 - Tension properties of cork specimens obtained from cork planks of different quality (class 1 and class 4) in the inner, outer and mid part of the plank. Mean of twelve samples and standard deviation

Compression		Class 1			Class 4	
properties *	Ab	Ai	Ac	Ab	Ai	Ac
E (MPa)	35.31±1.48	31.70±2.35	25.40±3.17	28.00±3.89	22.87 ± 4.50	26.65±3.18
$\sigma_{f}(MPa)$	1.27±0.07	1.00 ± 0.12	0.89±0.15	0.83±0.23	0.75 ± 0.14	0.73±0.10
ϵ_{f} (%)	8.22±0.68	6.27±1.36	6.89±1.0	5.72 ± 1.50	5.79±0.98	4.98±1.11
	Tb	Ti	Tc	Tb	Ti	Тс
E (MPa)	23.72±1.36	23.88±1.42	18.62±2.99	24.31±2.77	19.84±5.34	20.86 ± 5.00
$\sigma_{f}(MPa)$	0.75±0.10	0.62 ± 0.06	0.50±0.13	0.70±0.12	0.56 ± 0.20	0.57±0.13
ϵ_{f} (%)	5.97±1.11	4.50±0.63	4.44±1.44	4.82±0.92	4.40±0.83	4.73±1.07



Fig. 3 - Stress-strain curves for the bending tests of cork specimens obtained from cork planks of class 1and class 4 in the two directions.

Compression		Class 1			Class 4	
properties *	Rct	Rbt	At	Rct	Rbt	At
E (MPa)	16,25±1,26	17,66±1,47	17,88±2,43	15,34±1,28	16,72±2,10	16,86±3,82
σ _f (MPa)	1,18±0,22	1,13±0,22	1,29±0,11	0,98±0,07	1,3±0,03	1,30±0,27
ε _f (%)	15,04±2,29	12,57±1,71	13,08±1,56	11,19±2,63	15,03±1,11	13,35±1,58
	Rca	Rba	Та	Rca	Rba	Та
E (MPa)	21,52±3,13	22,51±1,68	21,76±2,09	21,09±3,49	19,29±2,11	18,69±1,34
σ _f (MPa)	1,67±0,27	1,57±0,32	1,63±0,17	1,35±0,05	1,53±0,27	1,43±0,11
ε _f (%)	17,56±1,75	18,41±3,92	17,58±1,94	12,68±4,81	14,97±2,20	18,81±4,65

Table 3 - Bending properties of cork specimens obtained from cork planks of different commercial quality (class 1 and class 4, respectively good and poor). Mean of twelve samples and standard deviation.

The comparison of compression tensile and bending behaviour of cork is shown in Fig. 4 in relation to the average Young's moduli for class 1 and 4. When the fracture zone on bending tests was near the inner part plank we compared with the tensile value in the same plane. The Young's moduli in compression are lowest than in tensile or bending. The highest resistance of cork is observed for the tensile stress. The behaviour of cork in bending seems to be influenced by the specimen zone that is submitted to compression, because the variation for bending and compression are very similar. If the fracture zone in the specimen submitted to the bending test was near the inner part of the plank or in the outer part is not correlated with the bending Young's moduli, but this is an important factor for the Young's moduli measured in tensile.

For class 4 corks, the tensile Young's moduli are higher than those in bending and compression. There was no correlation between Young's moduli measured in bending and compressions tests, as it was the case for class 1 corks. This can be explained by the higher percentage of defects in this poor quality cork planks, which showed a higher porosity and more frequent occurrence of sclerenchimatic cells.

For the fracture stress values in tension (Fig. 5), the previously observed relation is inverted, and the average values of the bending stress fracture are higher then the observed in tensile stress fracture. The variability observed in tensile tests is superior to that observed in bending. While the average value of the Young's modulus in bending was higher to the Young's modulus in tension, the tension that is necessary to fracture is lesser. Because in that test is necessary exceed too the resistant of the compression zone.



Fig. 4 – Variation of the average values of the Young's modulus in bending, tensile and compression tests for cork planks of class 1(left) and class 4 (right).

Fig. 6 represent for class 1 and class 4 the variation of average values of strain in bending testes and tensile, for the two quality class. The average values of the fracture strain in bending tests are superior to those observed in the tensile tests, independent of the quality class. A similar behaviour is observed between the average values of fracture strain in tension and the average values of the fracture strain in bending tests for quality class 1.

So we can conclude that the bending Young's modulus depends on the behaviour of the zone submitted in compression. The fracture stress result for a set of two stress (tensile and compression) and the fracture strain are well correlated with the tensile zone.



Figure 5 - Variation of the average stress values in bending and tensile tests for cork planks of class 1(left) and class 4 (right).



Figure 6 – Variation of the average strain values in bending and tensile tests for cork planks of class 1(left) and class 4 (right).

CONCLUSIONS

Cork shows some differences in its mechanical properties regarding the type of stress and the corresponding direction of stress. For the same direction of stress, the Young modulus in tension is higher then in bending and it is lowest in compression. The bending Young's moduli were well correlated with the tensile Young's moduli, because while in bending the sample is submitted to both tensile and compression stresses, the fracture occurs in the tensile zone. There were no significant differences in the mechanical properties of cork samples obtained from cork planks of different quality classes but the density is an important factor and samples with higher density showed overall larger resistance. Mechanical properties were influenced by the structural features related to the lenticular channels, namely the presence of thick walled and lignified cells that may border the lenticular channels.

The results showed that the cork resistance was higher for the inner part plank, caused by the cork structure. The cork was higher resistant too for the axial direction because in that direction there aren't so many residual tension. The mechanical behaviour is well correlated with the density and strongly affected by the cell structure defects namely the porosity.

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APPENDICE – Density and porosity of test specimen

Table A1 - Density and porosity of the cork specimens used in the compression tests obtained from cork planks of different commercial quality. Mean of twelve samples, standard deviation, and minimum and maximum values

Cork plank	Direction of	Density (g cm ⁻³)	Porosity * (%)
quality	compression	Mean±std.dev	Mean±std.dev
	Radial	0.152±0.009	6.56±1.72
Class 1	Axial	0.152±0.011	4.26±1.72
	Tangential	0.151±0.0136	3.45±2.86
	Radial	0.162±0.0155	8.76±4.61
Class 4	Axial	0.162±0.0052	4.54±1.32
	Tangential	0.160±0.0087	4.75±2.48

* Porosity of the faces perpendicular to the direction of compression

Table A2 - Density and porosity of the cork specimens used in the tensile tests obtained from cork planks of different commercial quality (class 1 and class 4). Mean of twelve samples and standard deviation.

Cork plank	Plane of	Density (g cm ⁻³)	Porosity * (%)	
quality	tensile	Mean±std.dev	Mean±std.dev	
	inner	0.178±0.010	3.51±0.41	
Class 1	mid	0.169 ± 0.007	5.22±0.63	
	outer	0.152 ± 0.005	6.35±0.79	
	inner	0.171±0.012	4.67±1.15	
Class 4	mid	0.148±0.011	5.76±1.23	
	outer	0.160±0.015	7.80±0.64	

* Porosity of the higher faces parallel to the direction of tension

Table A3 - Density and porosity of the cork specimens used in the bending tests obtained from cork planks of different commercial quality (class 1 and class 4, respectively good and poor). Mean of twelve samples, standard deviation, and minimum and maximum values

Cork plank	Samplo	Density (g cm ⁻³)	Porosity * (%)	
quality	Sample	Mean±std.dev	Mean±std.dev	
	Rct	0.177±0.010	3.71±1.42	
	Rbt	0.170±0.007	7.21±2.23	
Class 1	At	0.176±0.008	4.60±0.93	
	Rca	0.174±0.008	3.86±1.05	
	Rba	0.174±0.007	7.18±2.46	
	Та	0.174±0.007	5.07±0.35	
	Rct	0.194±0.017	6.02±1.29	
	Rbt	0.187±0.014	10.83±1.43	
	At	0.180±0.014	6.07±0.82	
CIA33 4	Rca	0.194±0.013	7.06±1.32	
	Rba	0.189±0.011	11.03±2.83	
	Та	0.183±0.009	6.81±0.61	

* Porosity of the higher faces perpendicular to the direction of bending