

Effect of quality, porosity and density on the compression properties of cork

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Published online: 28 May 2008
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Abstract The compression properties of cork were studied on samples obtained from cork planks of two commercial quality classes (good and poor quality), with densities ranging from 0.12–0.20 g cm⁻³ and porosities from 0.5 to 22.0%. The stress-strain curves were characterized by an elastic region up to approximately 5% strain, followed by a large plateau up to 60% strain caused by the progressive buckling of cell walls, and a steep stress increase for higher strains corresponding to cell collapse. The direction of compression was a highly significant factor of variation, with cork showing higher strength for the radial compression.

Density influenced compression and cork samples with higher density showed overall larger resistance to compression in the three directions. In the elastic region, an exponential model of Young's modulus in function of cork density could be adjusted.

The effect of porosity on compression was small and the stress-strain curves were similar regardless of the porosity of the samples, although there was a trend toward an overall increase of stress with porosity for higher strains. Porosity was characterised by a high variability in the anatomical fea-

tures of the lenticular filling material and the presence of collapsed and thick walled lignified cells. The inclusion of a porosity parameter for the modelling of the elastic modulus did not improve the prediction obtained with density-based models.

There was no significant difference in the compression properties of cork samples obtained from cork planks of good and poor quality classes.

Einfluss von Qualität, Porosität und Dichte auf die Druckeigenschaften von Kork

Zusammenfassung Untersucht wurden die Druckeigenschaften von Kork an Proben aus Material zweier handelsüblicher Qualitätsklassen (gute und schlechte Qualität), mit einer Dichte zwischen 0,12 und 0,20 g/cm³ und einer Porosität zwischen 0,5 und 22,0%. Die Spannungs-Dehnungs-Kurven weisen bis zu ca. 5% Dehnung einen elastischen Bereich auf, gefolgt von einem großen Bereich mit 60% Dehnung aufgrund des Ausbeulens der Zellwände, und einem anschließenden steilen Spannungsanstieg bei höheren Dehnungen aufgrund von Zellkollaps. Die Druckrichtung war ein maßgebender Faktor für die Streuung der Eigenschaften. Bei radialem Druck ergab sich dabei eine höhere Festigkeit.

Die Dichte hatte einen Einfluss auf die Druckeigenschaften. Korkproben mit einer höheren Dichte wiesen eine größere Druckfestigkeit in allen drei Richtungen auf. Im elastischen Bereich konnte der E-Modul als Funktion der Korkdichte durch eine Exponentialgleichung beschrieben werden.

Der Einfluss der Porosität auf die Druckeigenschaften war gering und die Spannungs-Dehnungs-Kurven wurden durch die Porosität der Proben kaum beeinflusst. Allerdings

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war bei höheren Dehnungen ein Trend zu insgesamt höherer Spannung mit zunehmender Porosität erkennbar. Die Porosität zeichnet sich durch eine hohe Variabilität der anatomischen Merkmale des Füllmaterials in den Lentizellen sowie des Vorhandenseins kollabierter und dickwandiger lignifizierter Zellen aus. Ein Modell zur Beschreibung des E-Moduls in Abhängigkeit der Dichte konnte durch die Hinzunahme eines Parameters für die Porosität nicht verbessert werden.

Die Qualitätsklassen des Korkmaterials hatten keinen signifikanten Einfluss auf die Druckeigenschaften.

1 Introduction

Cork is a natural material of cellular structure with an interesting set of properties, i.e., low density, very low permeability to liquids, large compressibility with dimensional recovery, low conductivity, chemical stability and durability (Pereira 2007). Well known applications of cork are as sealant of wine bottles and as insulation material.

The properties of cork result from the characteristics of its cellular structure, a highly ordered arrangement of small, hollow and non-communicating cells (Pereira et al. 1987), as well as of the chemical composition of the cell walls, with suberin as the main structural component (Pereira 1988). The biological process of cork formation as the outer bark of a tree species (the cork oak, *Quercus suber* L.) is responsible for the features of this tissue (Graça and Pereira 2004).

One of the most important characteristics of cork is the presence of lenticular channels that cross the cork planks in the tree stem along the radial direction linking the outside to the internal living tissues and allowing gas exchanges. These channels are a conspicuous feature of cork, appearing in the tangential section of cork as pores with a more or less circular form (Pereira et al. 1996). The lenticular channels contain a filling material of loosely bound cells of different chemical composition and are occasionally lined by heavily lignified cells.

The porosity of cork is the main quality parameter and it is used to grade the cork raw material. Good cork will have few and small 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).

The compression properties of cork are relevant for the performance in use of cork products, i.e., for cork stoppers in wine bottling and sealing. Description of cork under compression has already been made (Rosa and Fortes 1988b, Gibson et al. 1981). The stress-strain curves for cork are linear-elastic up to about 7% strain, at which point elastic collapse gives a rather horizontal plateau which extends

to nearly 70% strain when complete collapse of the cells causes the curve to rise steeply (Gibson and Ashby 1997). 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 1997) and cellular dimensions (Pereira et al. 1992).

However little is known about the effect of cork quality and of lenticular porosity on its compression properties, an aspect that has an obvious practical importance given the large price difference between corks of differing quality. This is the rationale behind this work on the influence of porosity measured by image analysis and of commercial quality on the mechanical properties of cork.

2 Material and methods

The material was sampled from raw cork planks collected at an industrial mill. Four planks of good quality (class 1) and four planks of poor quality (class 4) were taken after the water boiling operation and air drying that are usually applied to cork in the industrial processing.

The test specimens were cut from each cork plank as cubes with 20 mm of edge and equilibrated in the laboratory environment to 7% mean moisture content. The specimens were weighed and density was calculated.

Compression tests were made at a constant crosshead speed of 2 mm min^{-1} (strain rate of $2 \times 10^{-3} \text{ s}^{-1}$) up to a strain of 80%. The compression axis was, in different tests, parallel to each of the three tree principal directions: axial, radial and tangential. 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%. For each compression direction, three specimens from each plank were tested, totalling 12 per quality class.

Previous to compression the porosity of the specimen cubes was determined by image analysis on the faces perpendicular to the compression axis, e.g. tangential faces for a radial compression. The image was acquired at a 10X magnification using a colour video camera (3CCD) and analysed with the Pericolor-Matra software using image analysis and mathematical morphology methodological tools (Soile 1995). The porosity was reported as a porosity coefficient, in %, representing the area of pores divided by the total area, and calculated as the mean of the two faces measured in each sample.

The results were statistically analysed by analysis of variance using as fixed variation effects the quality class and the compression direction, and their interaction.

3 Results and discussion

The compression behaviour of the cork samples is shown in Fig. 1 by the stress-strain curves up to a strain of 80%, for compressions parallel to each of the three main directions and for the two quality grades tested. In all cases, the curves followed the known 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. This behaviour is common to all previously described studies involving the compression of cork samples (Rosa et al. 1990, Rosa and Fortes 1988b, Gibson et al. 1981).

The comparison between compression directions showed that cork strength in the radial direction was higher than in the other directions, especially for strains above 10%, and that the stress-strain curves for compression in the axial and tangential directions were very similar, as previously described (Rosa et al. 1990). For small strains up to about 5%, the strength in the radial and axial directions was similar and above the strength in the tangential section; the Young's moduli averaged 18.3 and 16.9 MPa for the radial and axial directions, respectively, 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).

3.1 Quality classes

The stress-strain curves for both quality classes were similar (Fig. 1). The most significant differences referred to somewhat higher stress values for the region of larger deformations, corresponding to strains above 50%.

Table 1 summarizes the compression properties of the cork samples of both quality classes regarding Young's modulus, stress values for different strains, as well as total compression energy up to 80% compression. Between-sample variation was high in all cases and a statistical comparison showed no significant differences of the mean values of the two quality classes.

An 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 with compression in the tangential direction significantly different from the other values.

These results suggest that there is no significant difference in the compression properties of samples taken from

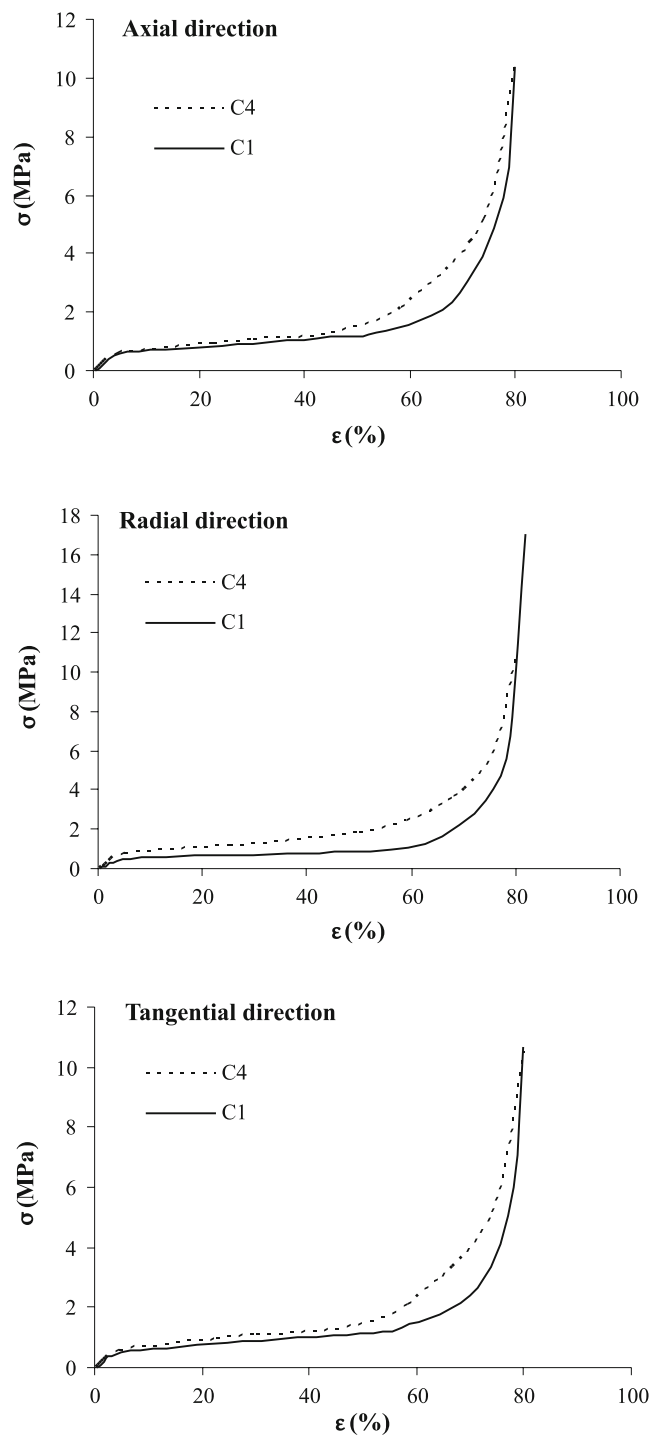


Fig. 1 Stress-strain curves for the compression of cork specimens obtained from cork planks of different commercial quality (class 1 and class 4, respectively good and poor quality) in the three directions (radial, axial and tangential)

Abb. 1 Spannungs-Dehnungs-Kurven druckbeanspruchter Korkproben der Qualitätsklassen C1 und C4 in den drei Richtungen (radial, axial, tangential)

cork planks of different commercial quality classes for the stress-strain values corresponding to their applications, e.g. strains between 25 to 50%.

Table 1 Compression properties of 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 properties* | Class 1 | | | Class 4 | | |
|---|--------------|--------------|--------------|--------------|--------------|--------------|
| | Radial | Axial | Tangential | Radial | Axial | Tangential |
| <i>E</i> (MPa) | 17.9 ± 2.86 | 16.6 ± 1.79 | 13.4 ± 1.42 | 18.6 ± 3.31 | 17.1 ± 2.27 | 11.2 ± 1.73 |
| σ_5 (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 |
| σ_{25} (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 |
| σ_{50} (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 |
| σ_{75} (MPa) | 5.34 ± 0.105 | 5.31 ± 0.163 | 5.37 ± 0.147 | 6.37 ± 2.35 | 6.97 ± 2.66 | 6.38 ± 0.389 |
| σ_{80} (MPa) | 18.0 ± 0.217 | 10.6 ± 0.330 | 10.7 ± 0.303 | 10.3 ± 0.271 | 10.4 ± 0.331 | 10.4 ± 2.29 |
| <i>U</i> 10 ⁻⁶ (Jm ⁻³) | 1.71 ± 0.042 | 1.36 ± 0.124 | 1.27 ± 0.106 | 1.83 ± 0.113 | 1.57 ± 0.041 | 1.48 ± 0.080 |

*Young's modulus (*E*), stress for strains of 5% (σ_5), 25% (σ_{25}), 50% (σ_{50}), 75% (σ_{75}) and 80% (σ_{80}), and total energy for 80% strain (*U*)

Tabelle 1 Druckeigenschaften von Proben aus Korkmaterial unterschiedlicher Qualitätsklassen (Klasse 1: gut, Klasse 4: schlecht) in den drei Richtungen (radial, axial und tangential). Mittelwert von zwölf Proben sowie Standardabweichung

The comparison of the density and porosity of the cork specimens used in the study (Table 2) showed a spread of values with a similar range in both quality classes. Since both characteristics are known to influence the compression properties of cork, an analysis of their individual effect is made next.

3.2 Density

Density influences the compressive properties of cellular materials (Gibson and Ashby 1997). 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 and Fortes 1988a).

In the present study, the densities of the cork samples ranged from 0.121 to 0.197 g cm⁻³. Figure 2 shows that there is a trend of increasing the Young's modulus *E* with the density, especially for compression in the radial direction. The effect was less marked for compression in the tangential direction. The correlation between *E* and density was for the radial, axial and tangential directions of compression $R = 0.820$ ($P = 0.001$), $R = 0.861$ ($P < 0.000$) and $R = 0.898$ ($P < 0.000$), respectively.

As regards the compression behaviour for higher strains, the high density corks (0.17–0.20 g cm⁻³) showed stress values above those of low density (0.12–0.15 g cm⁻³), and the crushing and collapse of the cells corresponding to the steep increase of stress started for lower strains (Fig. 3).

A model for the variation of the Young's modulus (*E*) with the cork density (ρ) has been proposed (Gibson and Ashby 1997) as an exponential function of *E*/*E_s* with ρ/ρ_s , where *E_s* is the modulus of the solid material of the cell walls with density ρ_s . The data obtained allowed to adjust the following models:

$$\begin{aligned} \text{Radial} \quad \frac{E}{E_s} &= 0.185 \left(\frac{\rho}{\rho_s} \right)^{2.24}, \quad \text{with } R^2 = 0.656, \\ \text{Axial} \quad \frac{E}{E_s} &= 0.027 \left(\frac{\rho}{\rho_s} \right)^{1.33}, \quad \text{with } R^2 = 0.751, \\ \text{Tangential} \quad \frac{E}{E_s} &= 0.014 \left(\frac{\rho}{\rho_s} \right)^{1.09}, \quad \text{with } R^2 = 0.821. \end{aligned}$$

These results are indicative of the positive variation of *E* with density, the worse correlation being obtained for the radial compression. The results showed that it was for compression in this direction that the variation of *E* was more

Table 2 Density and porosity of the cork specimens used in the compression 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 quality | Direction of compression | Density (g cm ⁻³) | | Porosity* (%) | |
|--------------------|--------------------------|-------------------------------|-------------|----------------|------------|
| | | Mean ± std.dev | Min.–Max. | Mean ± std.dev | Min.–Max. |
| Class 1 | Radial | 0.152 ± 0.009 | 0.138–0.161 | 6.56 ± 1.72 | 4.36–9.72 |
| | Axial | 0.152 ± 0.011 | 0.136–0.173 | 4.26 ± 1.72 | 2.35–7.80 |
| | Tangential | 0.151 ± 0.0136 | 0.121–0.171 | 3.45 ± 2.864 | 1.43–10.62 |
| Class 4 | Radial | 0.162 ± 0.0155 | 0.197–0.145 | 8.76 ± 4.61 | 4.56–22.49 |
| | Axial | 0.162 ± 0.0052 | 0.169–0.155 | 4.54 ± 1.32 | 2.47–7.02 |
| | Tangential | 0.160 ± 0.0087 | 0.148–0.175 | 4.75 ± 2.48 | 0.55–10.30 |

*Porosity of the faces perpendicular to the direction of compression

Tabelle 2 Dichte und Porosität der für die Druckprüfungen verwendeten Korkproben der Qualitätsklassen C1 und C4. Mittelwert von zwölf Proben, Standardabweichung sowie Kleinst- und Größtwerte

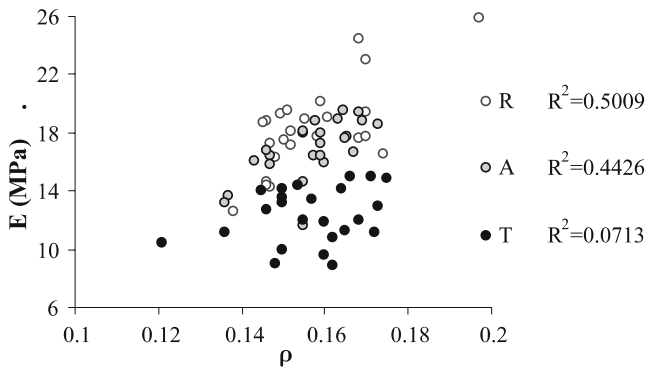


Fig. 2 Variation of the Young's modulus for compression in the three directions (radial R, axial A and tangential T) with cork density
Abb. 2 E-Modul in Abhängigkeit von der Korkdichte bei Druckbeanspruchung in den drei Richtungen (radial R, axial A und tangential T)

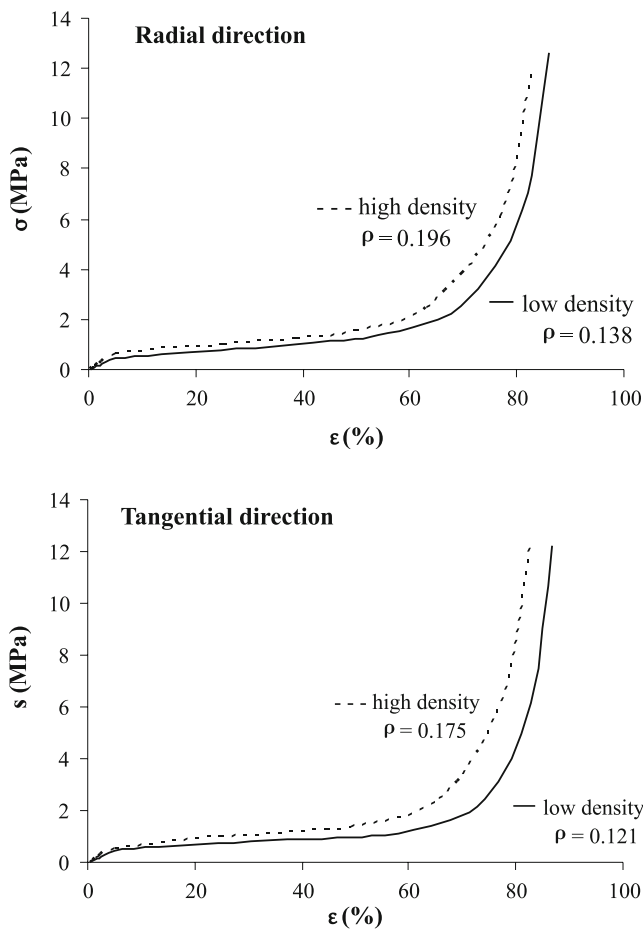


Fig. 3 Stress-strain curves for the compression of cork specimens with different densities (low density and high density) in the radial and tangential directions
Abb. 3 Spannungs-Dehnungs-Kurven druckbeanspruchter Korkproben mit niedriger und hoher Dichte in radialer und tangentialer Richtung

sensitive to density. This is contrary to a previous proposal (Gibson et al. 1982) of a nearly linear relation of *E* with density, thereby indicating that other factors in addition to

cell wall undulation and thickness are involved in the deformation process.

3.3 Porosity

The range of porosities of the tested samples was large, from a minimum value of 0.6% to a maximum of 22.5% (Table 2), and no significant differences between quality classes of the original cork planks could be detected on the samples. This is in accordance with the heterogeneity of porosity found in one cork plank that allows the possibility of extracting small samples, i.e., cork stoppers, with differing porosities. In fact, stoppers obtained from one cork plank are distributed along all the quality classes, the differences being on the absolute yields in each class, e.g. a good quality cork plank will yield more good stoppers (Pereira et al. 1994).

The stress-strain curves were similar regardless of the porosity of the samples. Figure 4 shows no influence of the porosity measured on the faces perpendicular to compression direction on the Young's moduli for compression in either direction. For higher strains, there was a trend for an overall increase of stress with porosity, as exemplified in Fig. 5 by the variation of the stress corresponding to a 25% strain with the porosity.

No studies are available to compare the influence of cork porosity on the compressive properties but some authors have reported that the effect of pores cannot be ignored and may be the explanation for some of the unaccounted differences between the samples (Pereira et al. 1992). However, the effect may be difficult to follow since several superposed effects might be involved due to the variability found in the anatomical characteristics of the cork porosity: the lenticular channels are filled with a non-suberous material, in some cases are lined by thick walled lignified cells, and vary in number and

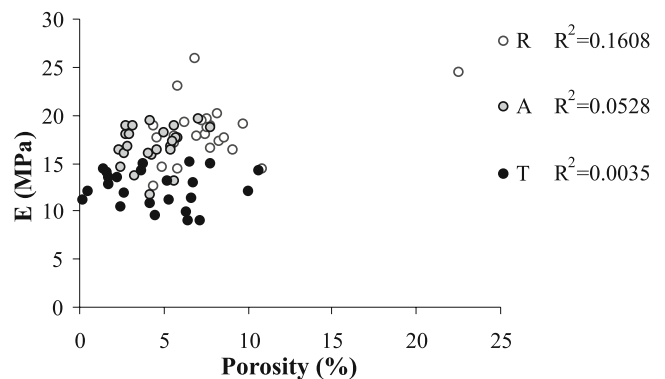


Fig. 4 Variation of the Young's modulus for compression in the three directions (radial, axial and tangential) with cork porosity measured on the faces perpendicular to the direction of compression
Abb. 4 E-Modul in Abhängigkeit von der Korkporosität bei Druckbeanspruchung in den drei Richtungen (radial, axial und tangential), die senkrecht zur Druckrichtung gemessen wurde

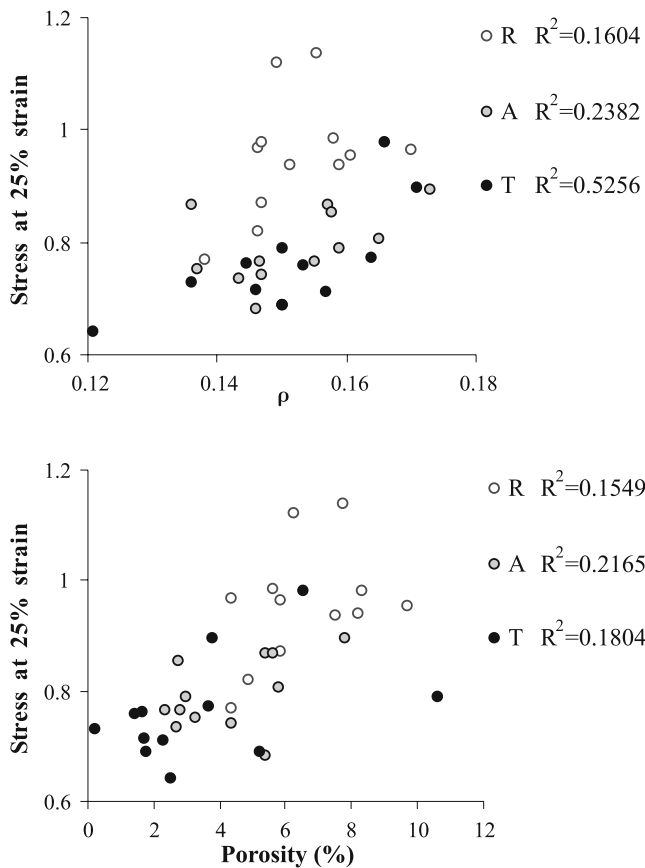


Fig. 5 Variation of the stress for a 25% strain for compression in the three directions (radial R, axial A, and tangential T) with cork density and porosity measured on the faces perpendicular to the direction of compression
Abb. 5 Druckspannung bei 25% Dehnung in den drei Richtungen (radial, axial und tangential) in Abhängigkeit von der Korkdichte und der Porosität, die jeweils senkrecht zur Druckrichtung gemessen wurden

dimensions (Pereira et al. 1996, Liese et al 1983). The porosity of cork is therefore not equivalent to voids, as it is in other materials, such as in wood. This is shown, for instance, by the lack of correlation between density and porosity found for all 72 cork samples used in this study (Fig. 6).

The observation of the cork by scanning microscopy confirmed these facts. First, the lenticular channels are filled with a material made up of rather spherical cells that although showing numerous intercellular voids are compactly arranged and have thicker cell walls than cork cells (Fig. 7a). In frequent cases, the cork cells in the region surrounding the pores were collapsed to some degree (Fig. 7b). The occurrence of lignified cells with thick walls as bordering cells of the lenticular channels is also frequent. These features will contribute to a higher density of the cork sample in the region of the lenticular channels and are the explanation for the increase of cork density with porosity (Fig. 6). The inclusion of a porosity parameter for modelling the elastic modulus, as proposed for wood (Young et al. 1998), is

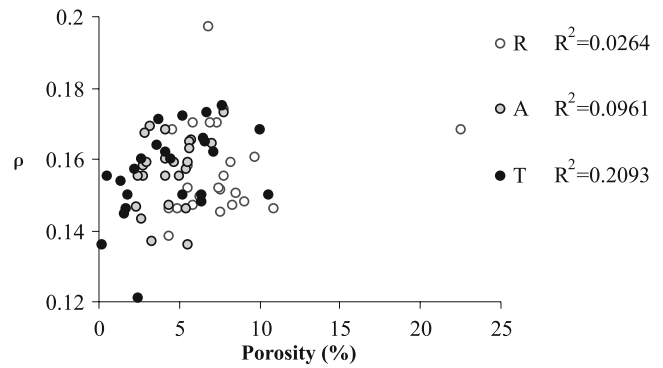


Fig. 6 Density and porosity of the cork samples measured on the faces perpendicular to the direction of compression
Abb. 6 Dichte und Porosität der Korkproben, die jeweils senkrecht zur Druckrichtung gemessen wurden

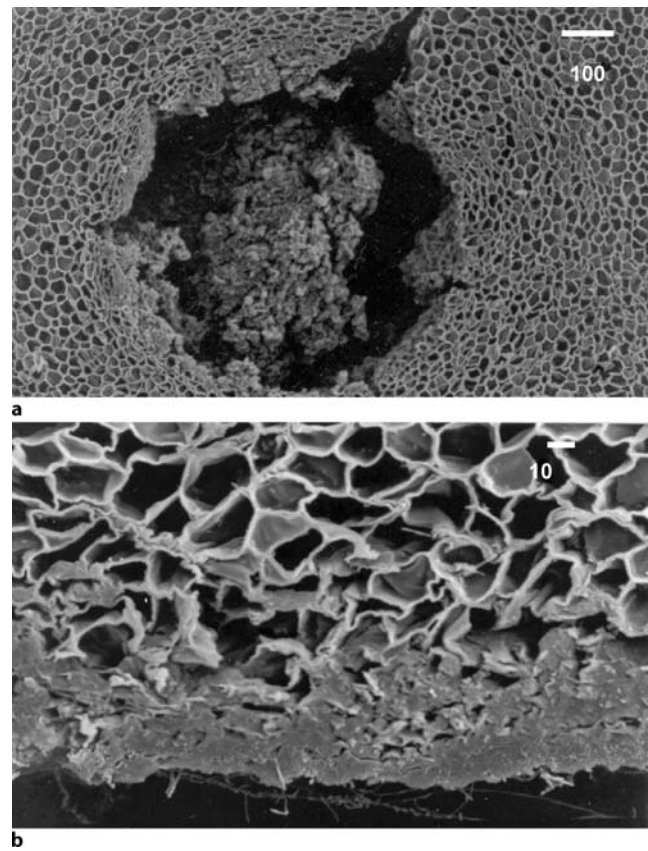


Fig. 7 Scanning electron micrographs of cork. **a** Filling material inside a lenticular channel observed on a tangential section **b** Structure deformation and cell collapse near a lenticular channel
Abb. 7 Rasterelektronenmikroskopaufnahme von Kork **a** Füllmaterial in einer Lentizelle in einem Tangentialschnitt **b** Strukturverformung und Zellkollaps an einer Lentizelle

therefore not adequate for cork and did not improve the prediction obtained using the density-based models.

Nevertheless the present results show that the compressive behaviour of cork samples with dimensions in the range

of those of stoppers is not influenced to a significant extent by the porosity values at the strain levels used in practice, i.e., stoppers in the wine bottle are compressed to about 30% of the initial diameter.

The large diversity of structural features found in the cork samples is responsible for the deviations found to the model predictions, especially in the case of the radial compression.

4 Conclusion

The compressive behaviour of cork samples of different commercial quality classes was similar and it was not influenced to a significant extent by the porosity values at the strain levels used in practice.

The variation in the compressive properties of cork could be associated with differences in density and cork samples with higher density showed overall larger resistance to compression in the three directions. In the elastic region an exponential model of Young's modulus in function of the cork density could be adjusted and the introduction of the porosity did not improve the model. Porosity was positively correlated with density and associated with structural features involving the lenticular channels.

Acknowledgement We thank Subercentro Cortiças, Lda. in Ponte de Sor for the cork planks and Pedro Pina for his help to develop the image analysis algorithm.

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