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Cork of Douglas-fir bark: Impact of structural and anatomical features on usage

Sofia Cardoso*, Joana Ferreira, Teresa Quilhó, Helena Pereira

Centro de Estudos Florestais, Instituto Superior de Agronomia, Universidade de Lisboa, Tapada da Ajuda 1349-017 Lisboa, Portugal

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ABSTRACT

Douglas-fir (*Pseudotsuga menziesii* (Mirb.) Franco) is one of the best conifer timber species. Its bark contains a substantial proportion of cork that may have a valorization potential, given adequate structural and cellular features. This study was made here on

bark samples from mature trees from the north and central mountains of Portugal. The area proportion of cork was determined by image analysis, the cork tissue was observed with electron scanning microscopy (SEM) and cell dimensions measured.

The cork is not continuous within the rhytidome, and the layers are interspersed with phloem regions. The cork layers are not continuous along the tangential or axial directions. Older trees contain on average a thicker rhytidome and a higher proportion of cork. The cork tissue was characterized by the presence of extensive areas of cells that are crushed or completely collapsed, making up a compressed and very compact structure with patches of uncompressed cork. The compression occurs in the radial direction and is clearly observed in transverse and radial sections. In the uncompressed regions the majority of the cork cells are hexagonal and pentagonal prisms stacked base-to-base and aligned in the radial direction in parallel rows. On average, the prism height and base area are 55 μm and 1388 μm^2, respectively, with a 1.3 μm cell wall thickness. To obtain pure cork fractions from Douglas-fir bark, trituration and fractionation processes are needed. Also the use of Douglas-fir cork as a cellular material will be restricted by the extensive cell compression.

1. Introduction

The cork that is stripped from the outer bark of the cork oak (*Quercus suber* L.) is one important European non-wood forest product and an economically relevant industrial raw material. The cork oak forests are geographically restricted to the western part of the Mediterranean with the largest production concentrated in Portugal and Spain (Pereira and Tomé, 2004). Cork has a closed cellular structure which together with the chemical features are the basis for its specific combination of properties: for instance low density, permeability and heat transfer, with large compressibility without fracture, and high durability (Pereira, 2015). The multiple applications of cork are recognized around the world: wine stoppers, cork composite materials e.g. insulation boards, surfacing materials, and other products (Pereira, 2007).

There are other tree species with barks that contain substantial amounts cork and some were characterized as potential cork providers: *Quercus cerris* (Turkey oak) (Şen et al., 2011a), *Quercus variabilis* (oriental or Chinese cork oak) (Miranda et al., 2013b), *Betula pendula* (Pinto et al., 2009) and *Kielmeyera coriacea* (Rios, 2011). The utilization of cork from the bark of Douglas-fir (*Pseudotsuga menziesii* (Mirb.) Franco) was also considered triggered by the large availability of this bark, namely in northern America (Hergert and Kurth, 1952; Krahmer and Wellons, 1973; Litvay and Krahmer, 1977).

Douglas-fir is one of the best timber conifer species, autochthonous to North America, and now also widely distributed in European forests: it occupies approximately 19 million ha in the USA and Canada (Weiskittel et al., 2012) and over 550 thousand ha in Europe. Douglas-fir provides knot-free sawn timber of great length and quality due to the large stem size and excellent wood properties. Most timber comes from plantation forests in Europe and North America (Ross and Krahmer, 1971).

Large quantities of Douglas-fir bark are therefore available as a residue from the primary processing of logs at sawmills or other processing mills. The potential of Douglas-fir bark was recognized

* Corresponding author.
E-mail addresses: sofiacardoso@isa.ulisboa.pt (S. Cardoso), jpferreira@isa.ulisboa.pt (J. Ferreira), terisantos@isa.ulisboa.pt (T. Quilhó), hpereira@isa.ulisboa.pt (H. Pereira).

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early and several studies were published, as compiled by Kurth (1950) and Hall (1971). One of the interesting features of Douglas-fir bark is that it contains a substantial proportion of cork in the rhytidome (Krahmer and Wellons, 1973; Patel, 1975).

The idea of commercially using the cork component of Douglas-fir bark is not new and was proposed in 1943 by Professor Bror Grondal (University of Washington) when cork was considered a vital war product, therefore potentially allowing the U.S. to become self-sufficient ("Eugene Register-Guard – Google News Archive Search," 1943). However this did not turn into reality even if some studies were continued (Laver and Fang, 1989; Graça and Pereira, 1999).

Douglas-fir bark structure is complex and cork is present in a substantial proportion. However the cork is not continuous within the rhytidome, and the layers are interspersed with phloem regions which makes cork separation difficult (Ferreira et al., 2015a). This is what occurs also in other cork containing barks with a similar rhytidome architecture, as for instance *Q. cerris* (Şen et al., 2011a). Although the structure of bark and cork of Douglas-fir was already described (Chang, 1954; Grillos, 1956; Hall, 1971; Hergert and Kurth, 1952; Litvay, 1976; Patel, 1975; Percival, 1948; Ross and Krahmer, 1971), it was not analyzed in detail from a material's point of view, namely regarding the cellular features of cork that would impact on its properties and product performance (Pereira, 2015).

This is the aim of the present study where the structure of the cork tissue in the bark of Douglas-fir was described in relation to topological arrangement, geometry and dimensions of the cells, and discussed regarding impact on properties and potential uses. A comparison is made with the cellular characteristics of cork from *Q. suber* which is the benchmark for this type of material. The objective is to contribute for the potential utilization of Douglas-fir bark residues through the valorization of its cork component.

2. Materials and methods

Bark samples of Douglas-fir (*Pseudotsuga menziesii* (Mirb.) Franco) were collected from a total of 20 trees in two sets (ten trees each) with approximately 44 and 100 years of age, respectively in the north and central mountains of Serra da Cabreira (41°35′18.0″N, 8°01′00.6″W) and Serra da Estrela (40°19′18.5″N, 7°36′49.8″W) in Portugal. The bark samples were collected from the bottom part of the trees, from the base up to 1.3 m of stem height. The bark samples were stored in indoor conditions with low light and good ventilation.

The transverse sections of the bark rhytidome were observed by image analysis and the area proportion of cork was calculated.

For electron scanning microscope (SEM) observations, small cubes with approximately 3 mm of edge were cut with a sharp razor blade in the cork region within the rhytidome. The cubes were mounted on stubs (ProSciTech, Australia) and sputter coated (Polaron E 5100 E, USA) with gold palladium for 3 min at 20 mA. The transverse, tangential and radial sections surfaces were observed in an SEM Hitachi S-2400 at magnifications ranging from 50 to 1000×, and the images were recorded in digital format.

In the SEM images corresponding to the different sections (transversal, tangential and radial) the cell measurements were made using an image analysis software (Leica Qwin Plus). The measurements were averaged for the tangential section (the honeycomb type cellular arrangement) and the non-tangential sections (the brick-wall type of structure) (Fig. 1). This type of measurements was only possible in the tissue regions where the cells were not corrugated or crushed, therefore representing the potential non-compressed cellular tissue. In the corrugated or crushed regions of cork, the measurements of individual cells cannot be made and only descriptive observations and general structural features are given.

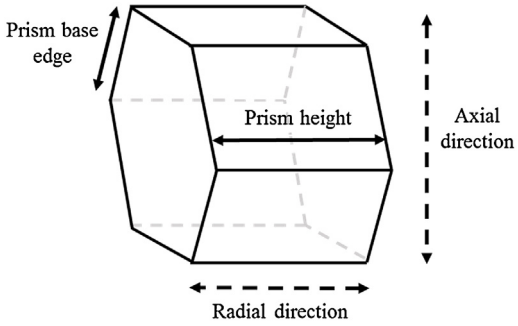

Fig. 1. Schematic drawing of a cork cell as a hexagonal prism showing the axial and radial directions and indicating where prism base edge and prism height were measured.

The number of edges of each cell i.e. the number of neighboring cells, was counted on the tangential and non-tangential sections. The distribution function of the number of edges of each cell was calculated based on the results of a total of 400 cells for each section as $f_i = N_i/\sum N_i$ where Ni represents the number of cells with i edges and $\sum N_i$ the total number of cells. The topological disorder of the bi-dimensional networks was evaluated by the dispersion of the function in relation to the mean (im) was calculated as $\mu_2 = \sum (i - i_m)^2 f_i$.

The average cell area was measured on the tangential sections, corresponding to the average prism base area, and the cell prism height was measured on the non-tangential sections. The cell wall thickness was measured in tangential and non-tangential sections as the radial dimension (Fig. 1). A total of 400 cells were measured for each section.

Fractionation of the Douglas fir rhytidome was made by using a cutting mill (RetschSM 2000) and a first pass with an output sieve with 10 mm × 10 mm openings, and a second pass with 6 mm × 6 mm openings, and sieved with a vibratory sieving apparatus (Retsch AS 200 basic) with standard sieves with mesh sizes of 80 (0.180 mm), 60 (0.250 mm), 40 (0.425 mm) and 20 (0.850 mm). The mass retained on each sieve was weighed and the corresponding yields were determined. Water flotation was applied to obtain cork fractions with higher purity after the laboratory scale fractioning. The fraction retained in the 20 mesh sieve was separated by flotation in distilled water with 24 h settling time after an initial mixing into a floating fraction of cork-enriched granules and a submerging fraction of phloem-enriched granules. Both fractions were separated, dried and weighed. Two replicates were made for the fractionation experiments.

3. Results

3.1. Rhytidome structure

The rhytidome of Douglas-fir bark comprises bands of phloem alternated with cork tissues visible to the naked eye: the cork has a light cream brown color and the phloem are dark brown (Fig. 2). The regions of cork are in general disposed as successive layers along the tree circumference but they are not continuous along the tangential or axial directions, and form a patchy pattern in the three sections. An estimate done by image analysis area measurement in the transverse section of bark pieces led to an average cork area proportion of 58% and 49% respectively in trees with 100 and 44 years of age; the older trees contained on average a higher proportion of cork although the range of variation was high. The radial width of the cork layers was also highly variable from about 1–4 mm.