J Wood Sci (2008) 54:464–469
DOI 10.1007/s10086-008-0972-6

ORIGINAL ARTICLE

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The influence of heartwood on the pulping properties of *Acacia melanoxylon* wood

Received: April 24, 2008 / Accepted: June 20, 2008 / Published online: September 4, 2008

Abstract The pulping wood quality of *Acacia melanoxylon* was evaluated in relation to the presence of heartwood. The sapwood and heartwood from 20 trees from four sites in Portugal were evaluated separately at 5% stem height level in terms of chemical composition and kraft pulping aptitude. Heartwood had more extractives than sapwood ranging from 7.4% to 9.5% and from 4.0% to 4.2%, respectively, and with a heartwood-to-sapwood ratio for extractives ranging from 1.9 to 2.3. The major component of heartwood extractives was made up of ethanol-soluble compounds (70% of total extractives). Lignin content was similar in sapwood and heartwood (21.5% and 20.7%, respectively) as well as the sugar composition. Site did not influence the chemical composition. Pulping heartwood differed from sapwood in chemical and optical terms: lower values of pulp yield (53% vs 56% respectively), higher kappa number (11 vs. 7), and lower brightness (28% vs 49%). *Acacia melanoxylon* wood showed an overall good pulping aptitude, but the presence of heartwood should be taken into account because it decreases the raw-material quality for pulping. Heartwood content should therefore be considered as a quality variable when using *A. melanoxylon* wood in pulp industries.

Key words *Acacia melanoxylon* · Heartwood · Sapwood · Extractives · Pulp yield

Introduction

Acacia is a genus of trees and shrubs belonging to the subfamily Mimosoideae of the family Fabaceae with over 1500 species and a large diversity of soil occupation.[1,2] *Acacia* spp. are originally from Tasmania, Australia, but are now widespread in different regions. Some were brought to Europe as ornamentals, to stabilize dunes, and for timber applications and tanning.[3] In Portugal, they were introduced in the mid-nineteenth century and several species, mostly *Acacia melanoxylon*, *Acacia dealbata*, and *Acacia longifolia*, are distributed over the country, especially in the Atlantic zone.[4] They are now considered as invaders due to their capacity for adaptation to low fertility soils, resistance, and high growth rate, which reduce development of autochthonous species.[5] Suggestions have been made for the use of acacia trees for timber and pulp, and as a strategic measure to avoid its uncontrolled spread.

Acacia trees are used for several purposes, including furniture, woodcraft products and pulp,[6] charcoal and firewood,[7] and gum extraction for food and pharmaceutical products.[8] They are considered as valuable forest species and commercial plantations have been established in Australia, New Zealand, China, South Africa, and Chile.[3,6]

One of the uses of acacia wood is pulping and several studies have shown its applicability with different processes such as kraft, soda-anthraquinone (soda-AQ), modified sulfite-AQ (ASA), and sulfite-AQ-methanol (ASAM). Pulp yield values range from 42% to 56%, kappa numbers between 11 and 22, and ISO brightness between 20% to 45%.[7,9,10–13] The kraft pulps from *A. melanoxylon* and *A. dealbata* showed good results regarding several properties, particularly in comparison with *Eucalyptus globulus* pulp,[9] as well as alkaline peroxide mechanical pulps.[14] According to Paavilainen,[15] the future main source of pulpwood in Asia will be fast-growing *Acacia* and *Eucalyptus* plantations.

When considering the pulping quality of tree stems, heartwood is an important variable, mainly because its high extractive content compared with sapwood leads to higher consumption of bleaching chemicals and lower pulp yield and brightness.[16–20] Heartwood is present in considerable proportion in tree stems, even in young pulpwood plantations of fast-growing species, such as eucalypts.[20–22] In *A. melanoxylon*, the heartwood amount is considerable, representing 61% of the total tree volume, and it seems to be

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influenced by environment rather than by genetic factors.[23,24] A number of secondary metabolites were reported for various *Acacia* species, the best known being gums and condensed tannins.[2]

An assessment of the pulpwood quality of *A. melanoxylon* trees should therefore include an evaluation of heartwood extractives and of their influence in pulping. This is the aim of the present study where extractives and their impact on pulp yield and properties were studied using heartwood and sapwood of different *A. melanoxylon* trees grown in four sites.

Materials and methods

Site characterization and sampling

The study was made with 20 trees of *Acacia melanoxylon* R. Br., with ages in the range of 28–43 years, from four locations in Portugal (5 trees from each site): site 1, Caminha; site 2, Ponte de Lima; site 3, Viseu; and site 4, Ovar. Geographic and edapho-climatic characterization was described elsewhere by Knapic et al.[23] The trees reached, on average, 30 m in height and 40 cm in breast height diameter. The heartwood attained 67% to 85% of tree total height and represented in cross section, on average, 69% at the stem base, decreasing to 26% at 75% of total height.[23]

Chemical analysis and pulping were performed on a 5-cm-thick disc collected at 5% of total height (corresponding to a mean height of 1.5 m), where the heartwood proportion ranged from 60% to 75% of the cross-sectional area. The sapwood and heartwood were separated on each disc with a chisel and milled in a knife mill (Retsch SM 2000), and sieved (Retsch ISO 9001). The 40–60 mesh granulometric fraction was kept for chemical analysis and the 20–40 mesh fraction for pulping.

Chemical analysis

The content of extractives was determined by using a Soxhlet extraction system with dichloromethane, ethanol, and water as solvents, as adapted from Tappi 12 os-75. Klason and acid-soluble lignin content were determined in extractive-free material following Tappi 222 om-02 and Tappi UM 250 methods, respectively. The carbohydrate composition was determined by gas-liquid chromatography with a method adapted from Tappi 249 cm-00. The hydrolyzed carbohydrates were derivatized as alditol acetates and separated by gas chromatography (GC; HP 5890A, Agilent) with flame-ionization detection (FID), helium as carrier gas (1 ml/min), and a fused silica capillary column S2330 (30 m × 0.32 mm i.d. × 0.20 μm film thickness). The column program temperature was 225°–250°C, with 5°C/min heating gradient, and the temperature of injector and detector was 250°C. For quantitative analysis, the GC was calibrated with pure reference compounds and inositol was used as an internal standard in each run.

Pulping

Pulping experiments were carried out with eight stem discs (two discs per site) selected as having the highest (H) and an average (M) value of heartwood extractives content. Kraft pulps were produced in 100-ml stainless steel autoclaves rotated (microdigesters) in an oil bath with temperature control. The 20–40 mesh fractions of heartwood and sapwood that were used had an average particle length of 1.79 mm and a thickness of 0.29 mm, as measured by image analysis. These dimensions are adequate to reduce the influence of chip size on the diffusion process during pulping and it can be assumed that the liquor penetration rate in the wood chips is infinite. The pulping conditions were: liquor-to-wood ratio (ml/g) 4.5:1; sulfidity 30%; active alkali 20% (as Na_2O); cooking temperature 170°C; time at temperature 90 min; heating time to temperature 5 min. The calculated *H*-factor was 1387 (*H*-factor in heat-up time: 9; *H*-factor in cooking: 1378). At the end of pulping, the pulps were defibrated in a standard pulp disintegrator, thoroughly washed with deionized water, air-dried overnight in a climate-controlled room (55% relative humidity and 25°C, as described in Tappi 402 os-70), transferred to an oven at 60°C (6 h), changed to 100°C (1 h), and weighed for yield determination. This procedure was previously tested to verify that the samples achieved a constant weight. All pulping experiments were replicated and if the pulp yields differed by more than 0.5 they were rejected and the process was repeated.

Pulp characterization

The *A. melanoxylon* pulps were extracted by dichloromethane and a solution (1:1) of ethanol and water using a Soxhlet extraction system to remove residuals of extractable material. Chemical characterization of pulps included the determination of residual lignin as kappa number measured automatically with a Titralab Radiometer Analytical equipment (Tappi 236 os-76), cellulose polymerization measured as intrinsic CED viscosity (SCAN-CM 15:88), and content of hexeneuronic acid groups (C_{hexA}) determined by ultraviolet (UV) spectrophotometry (Shimadzu, UV-160A) according to the method established by Chai et al.[25] using hydrolysis of 30 mg of pulp in a mercuric chloride (0.6%)–sodium acetate (0.7%) solution (10 ml). Pulp color was characterized by CIE $L^*a^*b^*$ scale and brightness (Tappi 452 om-02) with a Minolta CM-3630 spectrophotometer. Statistical analysis was conducted using SAS for Windows version 9.13 and a significance level of 0.05.

Results and discussion

Chemical composition

The analysis of sapwood and heartwood of the *Acacia melanoxylon* trees in the four sites is summarized in Table 1. The overall mean composition was 6.2% for total extractives and 21.0% for total lignin with 18.6% Klason lignin. The sugar composition is summarized in Table 2. Glucose and xylose are the main monosaccharide units, with values ranging respectively from 67.2% to 69.5% and from 22.5% to 26.1%. Together, arabinose and galactose represent about 2.6% of the total content of neutral sugars, and these sugar compositions agree with the general structure of hardwood hemicelluloses.[26,27]

There are very few published references on the chemical composition of *Acacia* wood. For *A. melanoxylon*, Santos et al.[9,28] reported 3.2% total extractives and 17.5% total lignin. For other *Acacia* species, Collins et al.[13] reported 5.8% total extractives, 21.7% Klason lignin, and 69.8% polysaccharides for *Acacia auriculiformis*; Santos et al.[9] reported 3.5% extractives and 18.2% Klason lignin for *Acacia dealbata*; and Neto et al.[11] and Pinto et al.[10] reported 4.5% ethanol/toluene extractives, 27.6% Klason lignin, and 61.0% polysaccharides for *Acacia mangium*.

In relation to other fast-growing pulp woods, for instance eucalypts, *A. melanoxylon* shows similar values of lignin content. For example, the lignin content of *Eucalyptus globulus* has been reported in the range of 20.9%–27.0%.[9,11,29,30]

Comparison of the heartwood and sapwood fractions of *A. melanoxylon* trees shows chemical differences in terms of the extractives content (Table 1). Heartwood had more extractives than sapwood (7.4%–9.5% and 4.0%–4.2%, respectively) and the heartwood-to-sapwood ratio for extractives varied from 1.9 to 2.3. This increase in extractives was mainly due to an accumulation of ethanol-soluble compounds in heartwood, where they represented more than 70% of the total extractives (40% in sapwood). The content of dichloromethane-soluble extractives was negligible (0.4%), while ethanol-soluble and water-soluble extractives reached an average of 5.9% and 1.8% in heartwood and 2.0% and 1.7% in sapwood, respectively. There was no difference between heartwood and sapwood regarding the structural components, with a mean lignin content of 20.7% in heartwood and 21.5% in sapwood and a very similar carbohydrate composition of hemicelluloses (Tables 1 and 2).

It is known that the higher content in extractives of heartwood results from an enrichment process that occurs during heartwood formation and is cumulative along the tree's lifetime.[31] Comparative studies of heartwood–sapwood extractives in other hardwood species have shown similar results: for instance, in *E. globulus* respectively 5.7% and 3.5%[22] and 4.6% and 2.6%,[32] and in the urograndis eucalypt hybrid 7.6% and 3.7%.[33]

Overall there was very little chemical variation between the *A. melanoxylon* trees in this study. The extractive content in sapwood and heartwood was not influenced by the sites where the trees had grown, as showed by a Student-Newman-Keuls ($\alpha = 0.05$) comparative test of means. The between-tree variation was also small and could not be ascertained to differences in tree dimensions, because only weak or no correlations were found between extractives in heartwood and sapwood and the corresponding cross-sectional area ($r = 0.38$; $P < 0.1$ and $r = 0.10$; not significant, respectively). Nevertheless, smaller heartwood diameter seemed to be associated to higher contents of total extractives (Fig. 1).

The visual distinction between sapwood and heartwood was quantified with color parameters (Fig. 2). Sapwood had higher luminosity (L^*) than heartwood (77 and 55, respectively) and lower a^* values (4.8 and 11.3, respectively), meaning that heartwood is redder, while b^* values were similar (20.4 and 22.4, respectively).

Table 1. Extractives and lignin content of sapwood and heartwood from *Acacia melanoxylon* grown in four different sites

Analysis	Sapwood				Heartwood			
	Site 1	Site 2	Site 3	Site 4	Site 1	Site 2	Site 3	Site 4
Total extractives	4.1 ± 0.3	4.0 ± 0.4	4.2 ± 0.5	4.1 ± 0.7	9.5 ± 1.2	7.4 ± 2.5	8.1 ± 3.9	7.6 ± 2.4
Dichloromethane	0.4 ± 0.3	0.3 ± 0.1	0.3 ± 0.1	0.4 ± 0.2	0.5 ± 0.1	0.3 ± 0.1	0.4 ± 0.1	0.3 ± 0.1
Ethanol	1.9 ± 0.3	2.0 ± 0.2	2.2 ± 0.4	1.9 ± 0.8	7.3 ± 1.2	5.3 ± 2.3	5.8 ± 4.9	5.3 ± 2.4
Water	1.7 ± 0.2	1.7 ± 0.2	1.7 ± 0.2	1.8 ± 0.2	1.7 ± 0.2	1.8 ± 0.3	1.9 ± 0.2	2.0 ± 0.3
Total lignin	21.0 ± 0.7	21.9 ± 0.5	22.2 ± 1.2	20.8 ± 0.8	20.5 ± 0.8	20.8 ± 07	20.9 ± 1.1	20.5 ± 1.6
Klason lignin	18.4 ± 0.6	19.3 ± 0.4	19.8 ± 0.6	18.1 ± 0.8	17.9 ± 0.9	18.2 ± 0.9	18.7 ± 0.8	17.9 ± 1.1
Soluble lignin	2.6 ± 0.7	2.6 ± 0.2	2.4 ± 0.6	2.8 ± 0.4	2.5 ± 0.4	2.7 ± 0.5	2.2 ± 0.4	2.6 ± 0.5

Data given as mean ± standard deviation ($n = 5$) as percentage of oven-dried wood

Table 2. Sugar composition of sapwood and heartwood from *A. melanoxylon* grown in four different sites

Sugar	Sapwood				Heartwood			
	Site 1	Site 2	Site 3	Site 4	Site 1	Site 2	Site 3	Site 4
Arabinose	1.9 ± 1.7	1.6 ± 0.5	1.6 ± 0.7	1.7 ± 0.6	1.1 ± 0.3	2.2 ± 1.9	1.3 ± 0.4	1.8 ± 0.8
Xylose	24.1 ± 1.9	25.2 ± 1.2	25.4 ± 2.3	23.3 ± 0.7	26.1 ± 0.4	24.6 ± 1.8	25.7 ± 1.9	22.5 ± 2.1
Mannose	5.0 ± 2.6	4.7 ± 0.6	4.7 ± 0.9	5.0 ± 1.5	4.1 ± 0.7	5.4 ± 1.8	3.8 ± 1.1	4.9 ± 1.6
Galactose	1.1 ± 0.3	1.0 ± 0.2	1.1 ± 0.3	1.1 ± 0.2	0.9 ± 0.5	0.8 ± 0.1	1.1 ± 0.3	1.2 ± 0.3
Glucose	67.9 ± 3.2	67.5 ± 2.0	67.2 ± 2.9	68.8 ± 2.1	67.8 ± 1.2	66.9 ± 2.4	68.2 ± 2.7	69.5 ± 2.6

Data given as mean ± standard deviation ($n = 5$) as percentage of total sugars

Pulp characterization

Table 3 summarizes the extractives content of the sapwood and heartwood fractions used for the pulping experiments and the resulting pulp yield, kappa number, and viscosity.

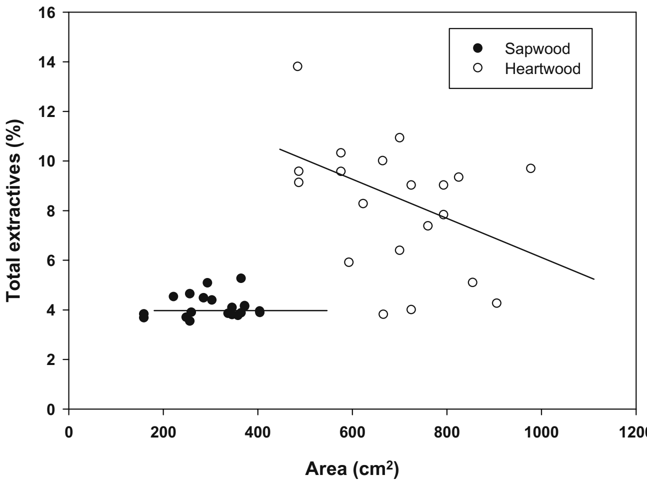

Fig. 1. Variation of total extractive content in heartwood and sapwood of *Acacia melanoxylon* trees grown in four sites with their respective cross-sectional area at 5% of stem height

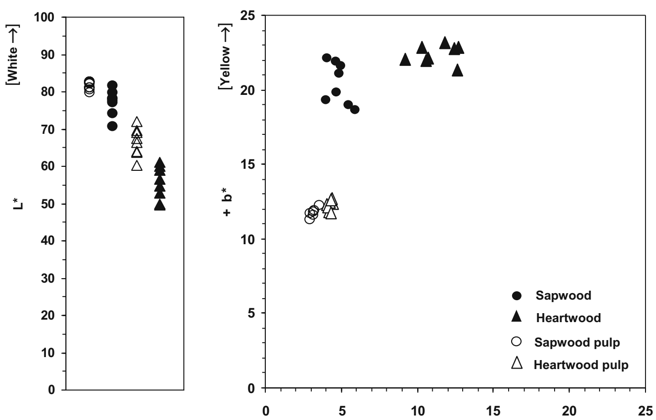

Fig. 2. Color values (CIE parameters $L^*a^*b^*$) of *A. melanoxylon* sapwood and heartwood (measured in 20–40 mesh samples) and of their unbleached pulps

The results of the kraft pulping showed an overall yield of 54.5% and low residual lignin (mean kappa number of 9). Similar pulp yield values of 55.7% and 53.2% with a kappa number of 11 were found by Gil et al.[34] and Santos et al.[28] using whole stems of *A. melanoxylon* as raw material.

Differences were found in the pulping of heartwood and sapwood (Table 3) with the pulp yield of heartwood 3.3% below that of sapwood. This is explained by the higher extractive content in the heartwood samples (9.2% vs 3.8% in sapwood). This negative influence of extractives in pulping was also reported by Miranda et al.[35,36] and Gominho and Pereira[37] for *E. globulus*.

The effect of heartwood on the delignification degree was small under the pulping conditions used, although heartwood pulps had somewhat higher kappa numbers (11 vs 7). Consequently, the effect on cellulose was also small and similar values were obtained for pulp viscosities (Table 3). However, the mean pulp viscosity found here was less than the values reported by Gil et al.[34] and Santos et al.[28] for the same species (1089 ml/g and 980 ml/g, respectively), suggesting that under the pulping conditions used, the cooking time was excessive and may require optimization for this wood material.

The content of hexeneuronic acid groups (C_{HexA}) was higher in heartwood pulps than in sapwood pulps (27–45 µmol/g and 11–29 µmol/g, respectively) (Fig. 3), but overall was below the values reported by Chai et al.[25] for hardwood pulps (39.6–69.3 µmol/g). Depending on the bleaching reagents and the sequence used, this fact can be an advantage for the bleachability of pulps obtained from *A. melanoxylon* wood because the presence of hexeneuronic acids in pulp generally has negative effects in bleaching and affects the kappa number determination.[38]

The brightness of heartwood pulps was on average 28%, considerably below the 49% brightness of sapwood pulps and showed little variation between samples (Fig. 4). The color parameters of the unbleached pulps are presented in Fig. 2. In relation to the original wood, the pulps showed a more uniform color with a reduction in a^* and b^* parameters and an increase in L^*. There were differences between heartwood and sapwood pulps that could be visually ascertained and that were quantified mostly by a lower lightness of heartwood pulps (66.6 vs 81.6) and of brightness (28% vs 49%) (Fig. 4). This lower brightness of heartwood pulps

Table 3. Total extractives in wood samples of *A. melanoxylon* used for pulping, unbleached pulp yields, kappa number, and pulp viscosity

Sample	Total extractives (%)		Total yield (%)		Kappa number		Viscosity (ml/g)	
	Sapwood	Heartwood	Sapwood	Heartwood	Sapwood	Heartwood	Sapwood	Heartwood
1H[a]	3.8	10.9	58.2	52.9	7	11	572	743
1M[a]	3.9	7.8	55.8	52.1	8	9	691	730
2H	3.7	9.7	55.6	53.1	7	10	729	757
2M	3.7	8.3	61.4	54.9	5	10	530	733
3H	3.9	13.8	53.6	49.6	9	14	729	870
3M	3.9	7.4	54.5	52.4	8	11	695	712
4H	3.5	9.6	56.6	55.2	6	10	651	708
4M	3.7	6.4	54.6	53.0	8	10	730	797
Mean	3.8 ± 0.1	9.2 ± 2.3	56.2 ± 2.5	52.9 ± 1.7	7 ± 1	11 ± 2	666 ± 77	756 ± 54

[a] Samples selected in sites 1 to 4 according to heartwood extractive content: H, highest; M, average

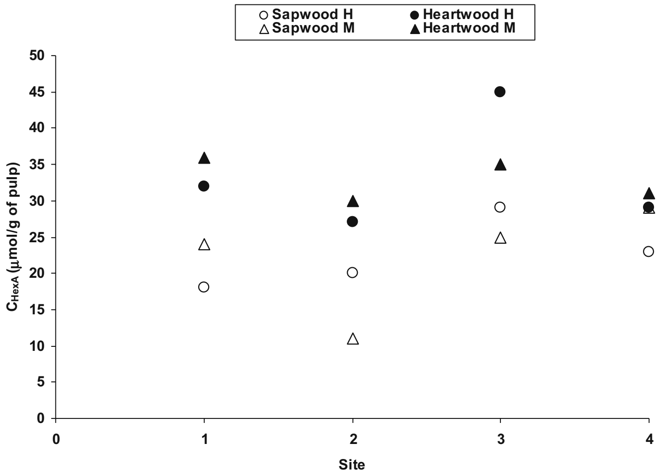

Fig. 3. Variation of hexeneuronic acid content in pulps obtained from sapwood and heartwood of *A. melanoxylon* trees grown in four sites. Samples selected in each site according to heartwood extractive content: *H*, highest; *M*, average

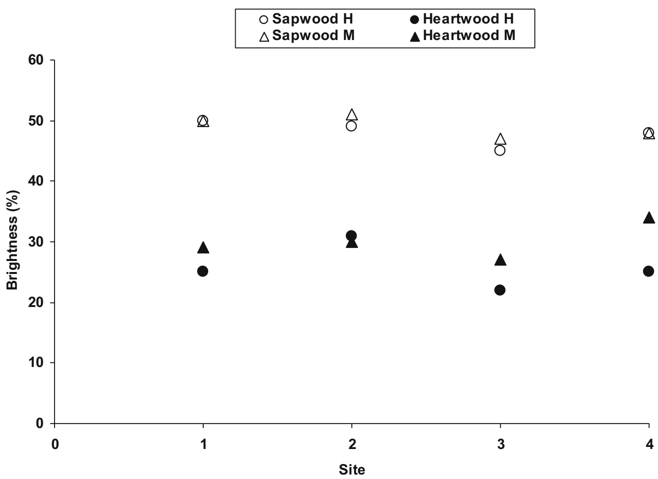

Fig. 4. Variation of brightness of pulps obtained from sapwood and heartwood of *A. melanoxylon* trees grown in four sites. Samples selected in each site according to heartwood extractive content: *H*, highest; *M*, average

has a negative impact because they result in higher consumption of bleaching chemicals. Gil et al.[34] reported 45% brightness for the same species, 37% for *Acacia longifolia*, and 41% for *A. dealbata*.

Conclusions

Acacia melanoxylon wood showed an overall good pulping aptitude in regard to chemical composition, with low lignin content leading to high kraft pulp yields and well-delignified pulps. There were chemical differences between heartwood and sapwood that influenced the pulping results. Heartwood contained on average twice the amount of extractives, while yield and brightness of heartwood pulps were lower than those of sapwood pulps. Considering that *A. melanoxylon* stems may contain a large proportion of heartwood, its content should be considered as a quality variable when using *A. melanoxylon* wood because its presence decreases the raw-material quality for pulp industries.

Acknowledgments The authors thank Fatima Tavares and Sofia Knapic, who supplied the material; Mamadú Camará and Joaquina Silva, who helped with chemical analysis; and Anna-Stiina Jääskeläinen for providing pulp samples for C_{HexA} calibration. Financial support was given by Fundação para a Ciência e Tecnologia (Portugal) through the programme Fundo Europeu de Desenvolvimento Regional/Programa Operacional "Ciência, Tecnologia, Inovação" (FEDER/POCTI) to Centro de Estudos Florestais and the project AGR/42594/2001.

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