Radial and Axial Variation of Heartwood Properties and Extractives in Mature Trees of *Eucalyptus globulus*

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Mature 40-year-old trees of *Eucalyptus globulus* harvested in Portugal were studied to determine the heartwood development and variation of basic density and extractives content at different stem height levels. The heartwood radius decreased regularly from bottom to top in all the trees: for instance 22.2 cm, 13.0 cm, and 10.4 cm, respectively, at the 0%, 35%, and 60% height levels of tree 1. The average sapwood thickness was 2.8 cm at the stem base. The mean basic density fell in a range between 0.607 g cm^{-3} and 0.782 g cm^{-3} and was highest in the outer heartwood at all height levels. The total content of extractives varied axially and radially along the stem. It decreased until the 35% height level, and then it remained approximately constant upwards. The extractives content increased radially from the sapwood to the inner heartwood (6.2% to 12.5% at the base). Ethanol-soluble compounds were the major fraction at the base: 4.9%, 8.4%, and 10.9% of dry mass, respectively, for sapwood, outer heartwood, and inner heartwood. The non-polar extractives were obtained by dichloromethane extraction in very low amounts.

Keywords: Eucalyptus globulus; Heartwood; Extractives content; Axial and radial variation

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INTRODUCTION

Eucalypt wood, in particular *Eucalyptus globulus* Labill. (Tasmania blue gum) is one of the main short-fiber feedstocks for the pulp and paper industries due to favorable biomass production and wood quality. *E. globulus* produces kraft pulps with high yields and quality for writing papers with excellent physical, optical, and printing properties (Pereira *et al*. 2010). The suitability of eucalypt wood fibers for pulp and paper is largely the result of cell biometry and raw-material chemical composition.

E. globulus wood has a high content of holocellulose (ranging from 50% to 69%), low extractives content (2.5% to 4.9%), and lignin content of 15.4% to 25.0% (Hillis 1984; Pereira 1988; Patt *et al*. 2006). The monomeric S/G composition of the lignin (1.9 to 2.3 using Py-GC/MS) also helps promote delignification (Lourenço *et al*. 2013a,b).

In spite of the low extractives content, their influence during the delignification and paper formation processes cannot be neglected, especially since the current pulp and paper mills adopt closed water circuits to meet environmental requirements (del Rio *et al*. 2000). The major negative impacts of extractives include the increase of alkali consumption during delignification, the decrease of pulp yield, and the enhancement of the formation of pitch (resinous material) and stickies, which cause serious operational problems (Gutiérrez *et al*. 2001).

Extractives are non-structural components of wood with high chemical diversity, *e.g.* hydrophilic and lipophilic compounds. Those compounds are responsible for the diversity of wood color, odor, taste, density, hygroscopicity, and durability (Fengel and Wegener 1989). Their occurrence, composition, and distribution depend on species, growing site, position within the tree, and genetic factors (Hillis 1984). In the tree, the extractives content varies axially and radially; it decreases towards the top of the tree and from the inner to the outer radius (Hillis 1980).

Heartwood is usually the part of the tree that accumulates extractives (Rowell 2005). In *E. globulus*, heartwood formation starts early (between 3 and 5 years of age) and represents a substantial portion of the stem volume, from 20% to 45% in 9-year-old trees (diameter at breast height, DBH, between 27.7 cm and 31.6 cm) (Gominho and Pereira 2000) and 55% to 65 % in 18-year-old trees (DBH between 13.1 cm and 15.2 cm) with (Miranda *et al*. 2006).

Sapwood extractives are different from heartwood extractives. Sapwood is rich in sugars related to the tree's physiological activity (*e.g.* glucose, fructose, and starch), in fatty acids (esterified to glycerol), and in small amounts of polyphenols, while heartwood is richer in polyphenols and resin acids (diterpenes). The soluble sugars contained in heartwood are mostly derived from the hydrolysis of hemicelluloses (*e.g.* xylose, mannose, and arabinose) due to the lower pH (Kai 1991).

For pulp and paper production, *E. globulus* trees are harvested in plantations at a young age: 9 to 12 years in Europe and 5 to 9 years in South America (Soares *et al*. 2007). At this age, also called the commercial age, the content of extractives and the presence of heartwood are lower, which minimizes their negative impact on pulping. Most of the research on *E. globulus* as a raw-material for pulp and paper deals with trees with such commercial ages, as reviewed in Pereira *et al*. (2010); however, there is a small amount of research on mature trees of *E. globulus* from old plantations that were not harvested and from other origins such as roadside lining trees. Due to the current raw-material shortage to feed the running pulp mills, this material has become a source of biomass for pulping. For such a type of wood, there is no information on the quality for pulping.

This work aims at covering this knowledge gap. Three mature *E. globulus* trees 40 years of age from an over-aged plantation were used as the raw material. Heartwood development and the radial (sapwood and two heartwood regions) and axial (at different levels of height) variations of basic density and extractives content were studied, with the objective of analyzing how an overaged eucalypt wood supply may affect industrial pulping.

EXPERIMENTAL

Materials

Three mature *Eucalyptus globulus* trees 40 years of age from one experimental plantation were harvested in Furadouro (Óbidos), Portugal. The site is located in the central coastal region, approximately 10 km from the Atlantic Ocean (39°20′N, 9°15′W, and altitude 30 m). The climate is of the Mediterranean type tempered by oceanic influence, with an annual rainfall of 607 mm and monthly mean maximum and minimum temperatures of 19.5 °C and 11.1 °C, respectively. The soil is eutric cambisols developed on sandstones. The plantation was established with plants from commercial seed source at 3 m x 3 m spacing.

In the harvested stems, 30 cm thick segments were taken at different height levels: 0%, 5%, 10%, 15%, 20%, 25%, 35%, 45%, 50%, 60%, 75%, of total tree height and top (6 cm diameter) (Fig. 1). The wood samples were taken to the laboratory and allowed to air dry (mean value RH = 60%). At the bottom part of each segment, two discs with 1.5 cm thicknesses were cut; one disc was used for stem cross-sectional determinations and the other for extractives analysis.

Methods

Stem determinations

In *E. globulus* the sapwood and heartwood are not always visually distinct; therefore the discs were stained with methyl orange at a 0.1% concentration for delimitation of heartwood, which colors red, *versus* sapwood, which colors yellow. The cross-sectional images were acquired using an image analysis system (AnalySIS® image processing software, Analysis Soft Imaging System, GmbH Münster, Germany; version 3.1) and the following areas were evaluated: total over-bark, total wood, sapwood, and heartwood. Heartwood diameters and sapwood radial thickness were calculated considering the areas to be circular.

The tree volume and heartwood volume were calculated as the sum of the volumes calculated for the different sections: as conical sections (between 0% and 75% of height) and cone (top level to total height) according to Gominho and Pereira (2000). The sapwood volume was calculated as the difference between the tree volume and heartwood volume. The merchantable bole was calculated as total volume less top volume.

The wood density was determined gravimetrically as basic density (g cm^{-3}) after water saturation according to TAPPI T258 om-02 (2002).

Extractives content

The heartwood and sapwood fractions were separated on each wood disc with a chisel, and the heartwood region was further divided into outer heartwood and inner heartwood at 1/2 of the heartwood radius (except for the segment at 75% of height, due to the small heartwood content). The samples were milled in a knife mill (Retsch SM 200; Germany), screened using a vibratory apparatus (Retsch AS 200), and the 40 to 60 mesh fractions were used for extractives determination. Only the levels of 0%, 5%, 10%, 35%, 50%, 60%, 75%, and top were analyzed.

The sapwood and heartwood samples (5 g) were Soxhlet extracted successively with dichloromethane, ethanol, and water until complete extraction (16 h with each solvent), and the extractives content was determined gravimetrically (adapted from TAPPI 204 om-88 1994). All the analyses were performed in duplicate.

RESULTS AND DISCUSSION

Tree Characterization

Figure 1 represents graphically the stem profiles and the sapwood and heartwood development along the height of the three harvested trees. The total tree height and over-bark diameter (at 1.30 m) were 29.1 m and 47.0 cm, 33.7 m and 49.2 cm, and 32.0 m and 32.0 cm, respectively, for Tree 1, Tree 2, and Tree 3. These *E. globulus* trees are larger when compared with the normal pulpwood supply to the industry, obtained from commercial plantations harvested at 9 years of age with a mean height of 18 m and 15 cm diameter at 1.30 m of height (Tomé *et al*. 2007); however, special attention must be taken at the mill woodyard since trees with large dimensions may generate problems during debarking and chipping.

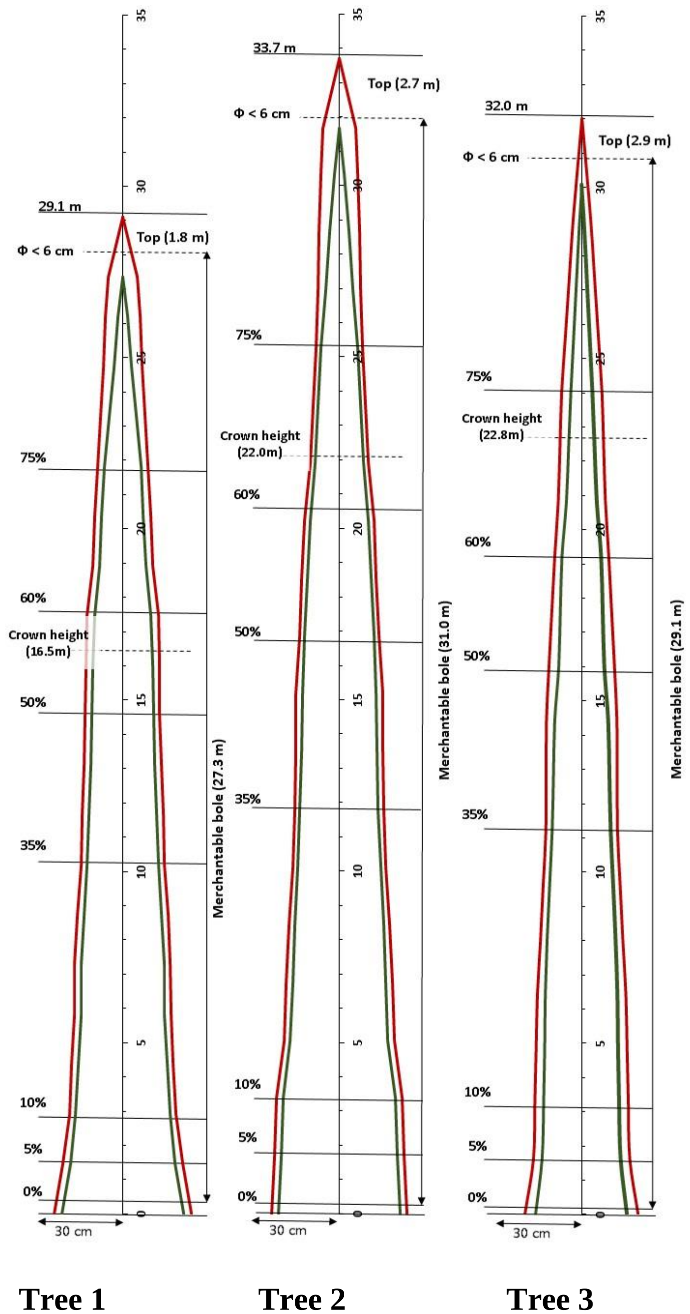

Fig. 1. Biometric data and heartwood and sapwood variation in the 40-year-old *E. globulus* trees harvested for this study (— sapwood; — heartwood)

Table 1 summarizes the biometric data of the trees at different height levels. Bark was thickest at the stem base (1.8 cm to 2.4 cm) and decreased along the height of the tree (0.3 cm to 0.5 cm at the top). These results are higher than the values reported for the *E. globulus* trees used in the pulp industry (Pereira *et al*. 2010), and reflect the older age of these trees. A similar pattern was found by Quilhó and Pereira (2001) in *E. globulus* of 15 years of age, with bark thickness decreasing from 1.0 cm at the base to 0.3 cm at the top.

The sapwood thickness was fairly constant along the height of the tree, and presented an average of 2.8 cm at the base level, 2.5 cm at 5% of height, and 2.3 cm at 60% of height. These values are similar to values reported for younger *E. globulus* trees. For instance, Gominho and Pereira (2005) presented values ranging from 1.5 cm to 1.7 cm at 5% of height and 2.6 cm to 3.0 cm at 65% of height in 9-year-old trees; Miranda *et al.* (2006) found 1.3 cm to 1.7 cm, and 2.0 cm to 2.5 cm for the same levels in 18-year-old trees, and for 9-year-old trees; and Monteoliva *et al.* (2002) reported sapwood thickness of 2.8 cm, 3.0 cm, and 1.9 cm at base, 30%, and 50% of height, respectively. The present results, which extend in tree age the available data on *E. globulus* sapwood thickness, strengthen the previous conclusions that sapwood thickness is fairly constant and species specific (Gominho and Pereira 2005; Pereira *et al*. 2010). In *E. globulus*, sapwood is about 2 to 3 cm thick.

Table 1. Biometric Data at Different Height Levels of the *E. globulus* Trees

Level (%)	0	5	10	35	50	60	75	Top
Tree 1								
Height level (m)	0.2	1.5	2.9	10.2	14.6	17.5	21.8	27.4
Total over-bark radius (cm)	27.0	23.5	21.1	16.3	14.3	13.7	9.6	5.7
Bark thickness (cm)	2.1	1.7	1.7	1.0	0.7	0.8	0.5	0.4
Sapwood thickness (cm)	2.7	2.7	2.2	2.3	2.4	2.6	2.4	2.7
Heartwood radius (cm)	22.2	19.1	17.3	13.0	11.2	10.4	6.7	0.0
Heartwood area (% of total area)	67.6	66.6	66.6	63.6	61.1	57.1	47.7	0.0
Tree 2								
Height level (m)	0.2	1.7	3.4	11.8	16.9	20.2	25.3	31.7
Total over-bark radius (cm)	26.4	24.6	24.0	16.2	15.2	13.3	9.0	6.5
Bark thickness (cm)	1.8	1.3	1.4	0.9	0.7	0.8	0.6	0.5
Sapwood thickness (cm)	2.6	2.3	2.4	2.1	1.9	2.0	1.7	3.0
Heartwood radius (cm)	22.0	21.0	20.2	14.1	12.5	10.6	6.7	0.0
Heartwood area (% of total area)	71.2	72.7	70.8	76.3	68.4	63.1	31.5	0.0
Tree 3								
Height level (m)	0.2	1.6	3.2	11.2	16.0	19.2	24.0	30.1
Total over-bark radius (cm)	19.1	15.9	15.2	11.5	10.6	8.7	6.5	2.0
Bark thickness (cm)	2.4	1.7	1.4	0.9	0.9	0.7	0.6	0.3
Sapwood thickness (cm)	3.1	2.5	2.6	2.8	2.9	2.1	2.7	0.9
Heartwood radius (cm)	13.6	11.7	11.2	8.7	6.8	5.9	3.3	0.0
Heartwood area (% of total area)	50.8	54.2	54.2	57.8	41.4	45.5	24.8	0.0

In all the trees, the heartwood radius decreased regularly from bottom to top. For instance, in Tree 1 the heartwood radius was 22.2 cm, 13.0 cm, and 10.4 cm, respectively, at 0%, 35%, and 60% of height; in Tree 3 these values were 13.6 cm, 8.7 cm, and 5.9 cm. This pattern of heartwood development has been reported by several investigators on eucalypt trees (Wilkins 1991; Gominho and Pereira 2000; 2005; Miranda *et al.* 2007). Gominho and Pereira (2005) observed that heartwood radius positively correlates with tree radius at different heights.

Heartwood represented more than 50% of the sectional area at the base, decreasing upwards; *e.g.*, at level 75% it represented between 24.8% in Tree 3 (the smallest tree) and 47.7% in Tree 1 (the largest tree). This is consistent with previous findings showing that heartwood development is in relation with tree diameter (Gominho and Pereira 2005). The tree volumes are summarized in Table 2. Heartwood represented in these mature trees account for a high content of total volume: on average 62%, with 60%, 67%, and 55% of the total volume respectively for Tree 1, Tree 2, and Tree 3. Heartwood formation is a cumulative process, and its volume in the tree increases with age and tree diameter; therefore the heartwood proportion of these 40-year-old trees was substantially above the values in *E. globulus* at a commercial age (9 to 14 year-old), where the heartwood represented 20% to 40% of the total wood volume (Gominho and Pereira 2000; Morais and Pereira 2007). Sapwood accounted on average for 30% of the tree volume and bark represented on average 13% of the total volume. Similar values for bark volumes were reported by Easwarankutty *et al.* (1977) in eucalypt trees. The higher content in heartwood in these mature trees of *E. globulus* is negative for pulping production as demonstrated by Lourenço *et al*. (2010), who found a difference of 4% in pulp yield when sapwood and heartwood from *E. globulus* were delignified separately.

Table 2. Total Volume and Partitioning in Bark, Sapwood, and Heartwood of the *E. globulus* Trees

	Tree 1	Tree 2	Tree 3	Mean
Total volume (m^3)	2.14	2.71	1.11	1.99
Bark volume (m^3)	0.28	0.30	0.20	0.26
Sapwood volume (m^3)	0.57	0.59	0.35	0.51
Heartwood volume (m^3)	1.29	1.82	0.56	1.22

Basic Density

The variation of basic density along the tree height in the sapwood and heartwood (outer and inner) regions is represented in Fig. 2 as mean values for the three trees; basic density ranged between 0.607 g cm^{-3} (sapwood) and 0.782 g cm^{-3} (outer heartwood) at the base. In height, the basic density practically remained constant in the sapwood, decreased in the outer heartwood, and slightly increased in the inner heartwood. The outer heartwood presented a higher basic density, especially in the lower part of the stem, contrary to what was shown in other works.

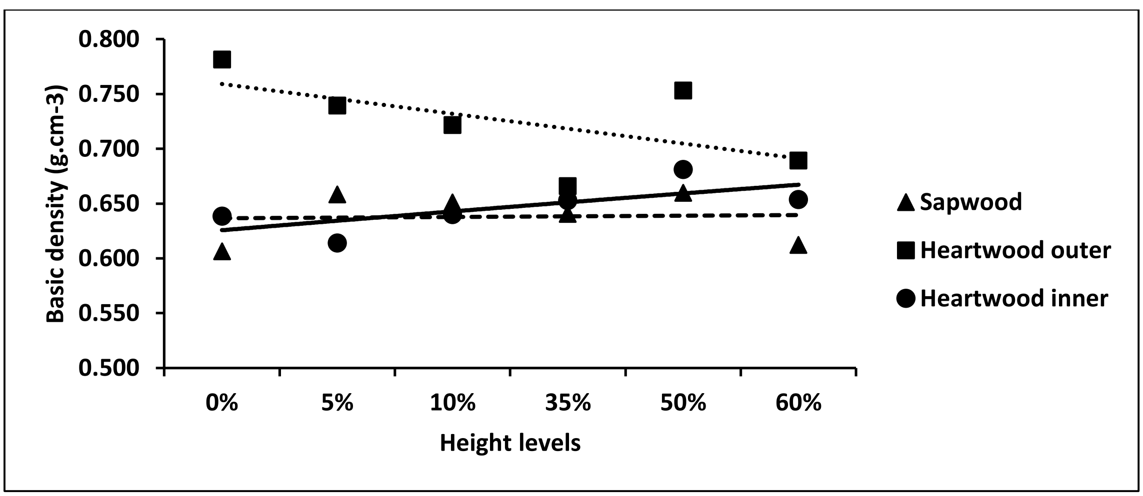

Fig. 2. Variation of basic density of sapwood and heartwood along the height levels of *E. globulus* trees. Mean values of the three trees are shown.

Wilkes (1988) for *E. globulus,* Bhat *et al.* (1990) for *E. grandis,* and Gominho *et al.* (2001) for *E. urograndis* (*E. grandis* x *E. urophylla*) reported that basic density increased in the direction of the bark. This variation may be explained by the combined and contradictory effect of age and accumulation of extractives; cell-wall will increase radially with cambial age (Rowell 2005) while extractives accumulate in the heartwood.

The values for the basic density found in this study were higher than *E. globulus* harvested at 7 years of age in other studies: 0.490 g cm^{-3} to 0.609 g cm^{-3} (Montoliva *et al.* 2002) and 0.540 g cm^{-3} to 0.564 g cm^{-3} (Quilhó and Pereira 2001). A high wood density is considered favorable by the pulp industry since it corresponds to more wood mass per volume in the digesters (Rydholm 1965; Hakkila 1989).

Extractives Variation

The extractives variation in the sapwood and heartwood along the different height levels is summarized in Table 3 as the mean for the three trees. Heartwood consistently contained more extractives than sapwood, principally the inner part of the heartwood. At the base height level (0%), the total extractives content represented 12.5%, 10.8%, and 6.2% of the dry mass of the wood, respectively, for heartwood $_{inner}$, heartwood $_{outer}$, and sapwood. The major part of extractives is made up of ethanol-soluble compounds, followed by the water-soluble components and the dichloromethane extracts. The amounts of extractives in this study were higher in comparison to values reported by Miranda *et al.* (2007) for 8-year-old *E. globulus*, but similar to those found in 36-year-old stumpwood by Gominho *et al.* (2012; 2014). This high accumulation of extractives in the mature *E. globulus* trees can be explained by the combined effects of age, growth, and heartwood formation.

Table 3. Variation of the Extractives Content (% of oven dry mass) in Sapwood and Heartwood along the Height Levels in the *E. globulus* Trees (Mean of the three trees ± Standard Deviation)

Level (%)	0	5	10	35	50	60	75	Top
Total								
Sapwood	6.2±1.19	4.9±1.15	4.9±1.66	3.7±0.36	4.0±1.62	3.3±1.00	5.7±0.31	5.2±0.32
Heartwood $_{outer}$	10.8±2.34	6.8±1.79	5.3±2.27	4.2±1.44	4.4±2.00	4.0±1.32	4.3±0.30	-
Heartwood $_{inner}$	12.5±2.19	9.4±4.45	7.9±2.46	4.2±0.95	4.4±1.36	4.8±1.26	-	-
Dichloromethane								
Sapwood	0.3±0.03	0.3±0.04	0.3±0.04	0.3±0.03	0.2±0.04	0.2±0.02	0.4±0.07	0.5±0.02
Heartwood $_{outer}$	0.4±0.05	0.4±0.04	0.3±0.01	0.3±0.09	0.3±0.06	0.3±0.05	0.4±0.05	-
Heartwood $_{inner}$	0.5±0.06	0.4±0.02	0.3±0.04	0.3±0.07	0.3±0.08	0.3±0.03	-	-
Ethanol								
Sapwood	4.9±1.42	3.4±0.93	3.5±1.29	2.3±1.28	2.9±1.82	1.9±0.76	3.9±0.20	3.0±0.15
Heartwood $_{outer}$	8.4±2.12	4.6±1.66	3.4±1.60	2.8±1.29	3.0±1.56	2.7±1.02	2.7±0.17	-
Heartwood $_{inner}$	10.9±1.84	7.7±4.27	6.3±2.38	2.7±0.76	3.2±1.20	3.6±1.06	-	-
Water								
Sapwood	1.0±0.37	1.2±0.18	1.1±0.50	1.1±0.13	0.9±0.18	1.2±0.30	1.4±0.15	1.8±0.20
Heartwood $_{outer}$	1.9±0.51	1.8±0.61	1.6±0.68	1.1±0.21	1.1±0.41	1.1±0.44	1.2±0.14	-
Heartwood $_{inner}$	1.1±0.36	1.3±0.24	1.2±0.33	1.1±0.38	0.9±0.19	0.9±0.22	-	-

- denotes absence of heartwood

The content of extractives varied axially and radially within the tree. Axially, the amount of extractives decreased until the 35% level, remained slightly constant up to level 60%, and then increased afterwards corresponding to the presence of the crown. Radially, the content of total extractives always increased to the inner part of the tree. The ethanol and water extracts showed the same pattern of variation, while the dichloromethane extracts exhibited little axial and radial variation. Such a high content in extractives in this pulpwood material should not be ignored, and special attention must be considered due to the negative impacts in pulp and paper mills (Hakkila 1989). Miranda *et al*. (2007) and Lourenço *et al*. (2010) demonstrated with *E. globulus* as raw-material that pulp yields negatively correlate with extractives content.

Table 4 reports the mass of wood in the merchantable bole (without bark). The sapwood represents 25.8% and the heartwood 74.2% of the total wood mass (35.2% in the outer heartwood region and 39.0% in the inner heartwood region). Considering the negative impact of extractives in pulp and paper production, the use of these mature trees as feedstock involves the necessity of dealing with an average of 76 kg of extractives per ton of pulpwood (3.5 kg of non-polar compounds, 58 kg of ethanol-soluble compounds, and 14 kg of water-soluble compounds). In terms of the contribution of the different wood regions to these amounts of extractives, the heartwood $_{outer}$ contributes to 50%, the heartwood $_{inner}$ to 33%, and the sapwood to 17% of the total extractives (Fig. 3).

Table 4. Mass of Oven Dry Wood in the Merchantable Bole Without Bark of the Harvested *E. globulus* Trees and Partitioning in Sapwood and Heartwood

Mass of wood (kg)	**Tree 1**	**Tree 2**	**Tree 3**	**Average**
Merchantable bole	1313	1420	618	1117
Sapwood	357	279	227	288
Heartwood $_{outer}$	507	594	210	437
Heartwood $_{inner}$	449	548	181	393

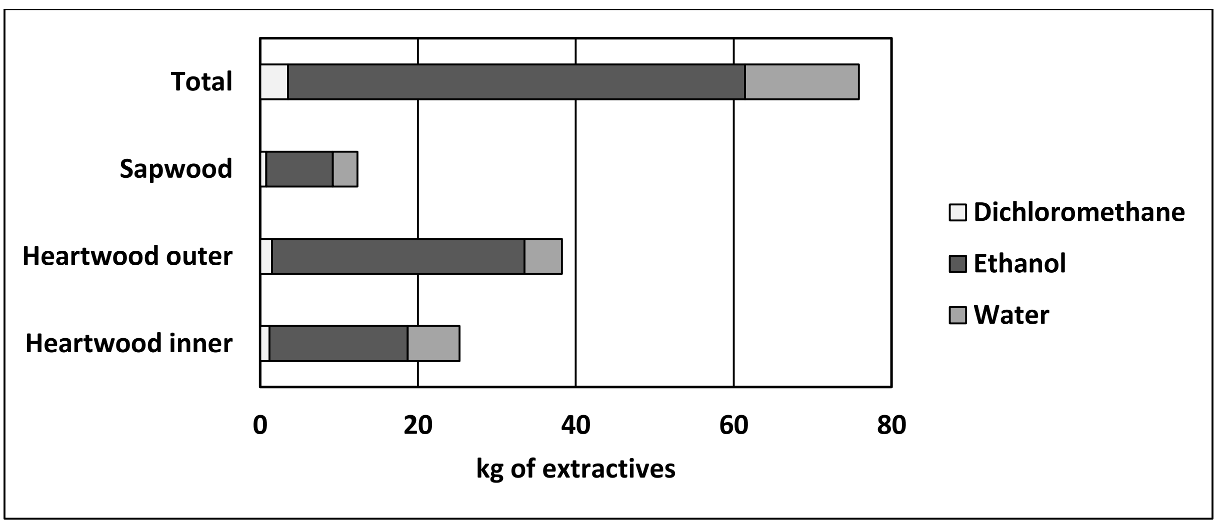

Fig. 3. Mean mass of extractives in the sapwood and heartwood of the merchantable bole of *E. globulus* trees

CONCLUSIONS

1. Mature *E. globulus* trees presented a higher content of heartwood and extractives, and a higher wood density than the commercial stemwood used for pulping.
2. The heartwood development followed the known decreasing pattern from the base of the tree upwards, while the sapwood thickness remained practically constant.
3. The heartwood always contained more extractives than the sapwood, principally in the inner heartwood. A higher content in extractives was found in the lower part of the stem. The extractives were mostly polar compounds, which could be extracted by ethanol and water, whereas non-polar extractives corresponded to a small proportion.
4. The industrial use of *E. globulus* mature trees as pulpwood should take into account that heartwood represents on average 62% of the wood supply mass and that extractives amount to 7.6% of the wood supply mass, which can negatively affect pulp and paper production.

ACKNOWLEDGMENTS

The authors are thankful for the support of the Portuguese pulp producer ALTRI and Clara Araújo for material supply. This work was supported by a FCT - Fundação para a Ciência e a Tecnologia project (PTDC/AGRCFL/110419/2009). Centro de Estudos Florestais (CEF) is a research unit supported by the national funding of FCT - (PEst-OE/AGR/UI0239/2014).

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Article submitted: October 8, 2014; Peer review completed: November 21, 2014; Revised version received and accepted: November 25, 2014; Published: December 4, 2014.