# Variation of Wood Pulping and Bleached Pulp Properties Along the Stem in Mature *Eucalyptus globulus* Trees

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The wood of a mature (40-year-old) *Eucalyptus globulus* Labill tree was characterized at different stem height levels (0%, 10%, 35%, and 50% of total height) regarding pulping, bleaching, and paper properties. Pulp yields increased upwards from 46% to 50%, and Kappa number decreased from 17.5 to 12.3 at 0 and 50% height, respectively. The estimated specific wood consumption ranged from 3.2 $m^3$ $odt^{-1}$ to 3.1 $m^3$ $odt^{-1}$ at 0% and 50% height levels, respectively. Pulp drainage varied along the stem, with less drainability (20.3 ºSR) and higher water retention value (1.07 $g.g^{-1}$) at the base. Pulp fiber length increased (827 µm *vs*. 877 µm) and width decreased (19 µm *vs.* 17 µm) from 0% to 50% height levels. Tensile, tear, and internal bond strength decreased upwards, with mean values of 34.9 $N.m.g^{-1}$, 3.1 $mN.m^2.g^{-1}$, and 95.8 $J.m^{-2}$, respectively. These findings support the use of mature *E. globulus* trees without loss of pulp production and quality.

*Keywords: Eucalyptus globulus; Bleaching; Pulping; Pulp properties; Papermaking potential*

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## INTRODUCTION

*Eucalyptus globulus* Labill. is one of the main short-fiber woods used by the pulping industry. It is highly appreciated for production of kraft pulps for printing and writing paper grades due to its excellent physical, optical, and printing properties (Pereira *et al*. 2010).

In Europe, commercial *E. globulus* plantations are found mainly in Portugal with 812 million ha (ICNF 2013) and Spain with 760 million ha (MAAMA 2013). Such plantations frequently are managed as a coppice system with 3 to 4 rotations of about 9 to 12 years each (Soares *et al*. 2007). However, mature *E. globulus* trees well above this cutting age are becoming more usual in the wood-yards of the pulping industries. These mature *E. globulus* trees coming from old plantations that were not harvested and from other origins such as roadside lining trees.

Such mature trees differ in fiber morphology, wood density, heartwood content, and, therefore, in the chemical composition, which may influence the pulping quality. In fact, fiber length and wall thickness increase with age in *Eucalyptus* species (Wilkes 1988). Basic density also increases with age as a result of the thicker fibers and of more heartwood (Gominho and Pereira 2000; Gominho *et al*. 2001). For instance, Gominho *et al*. (2015a) found values of 0.607 $g.cm^{-3}$ to 0.782 $g.cm^{-3}$ along the radial and axial directions in mature (40-year-old) *E. globulus* trees, while in wood with the usual cutting age, the values range between 0.467 g cm$^{-3}$ to 0.600 g cm$^{-3}$ (Santos *et al.* 2008). Another consequence of age is the increase in extractives, due to their accumulation in heartwood (Hillis 1980). For example, in *E. globulus* mature trees, heartwood represents 62% of the total volume and has a higher extractives content (Gominho *et al.* 2015a); Wiemmer *et al*. (2002) observed more cellulose and less lignin content.

Tree age therefore impacts the pulping and pulp properties: negatively, by loss of cell permeability, increase in alkali consumption, and promotion of pitch and stickies formation as a result of the presence of extractives (Tuner *et al*. 1983; Gutiérrez *et al*. 2001); and positively, by a lower specific wood consumption, higher coarseness, and low fiber flexibility (Santos *et al.* 2008). An exploratory study with large-sized material collected at the wood-yard of a pulp mill showed that over-aged material yielded bleached pulps with good properties, in spite of a lower pulp yield due to the higher content in extractives (Gominho *et al.* 2015b).

Mature trees have a higher within stem variation, both radially as well as axially along the tree, therefore leading to heterogeneities of behavior of different parts of the tree, *i.e.*, corresponding to different cambial ages that may influence the delignification and the pulp properties. In this work, we studied the axial variation of pulping and of the bleached pulp properties using three mature *E. globulus* trees from one over-aged plantation and analyzed the corresponding range of variation.

## EXPERIMENTAL

### Materials

Three mature *Eucalyptus globulus* trees with 40 years of age were harvested in Furadouro (Óbidos), Portugal. Stem wood disc samples (30 cm thick) were cut at different height levels of the stem (at 0, 10, 35, and 50% of total tree height) and taken to the storeroom to air dry (RH = 35%) (Gominho *et al*. 2015a). At the bottom part of each section, discs with 10 cm thickness were cut and milled in a knife mill (Retsch SM 2000) passing through a sieve of 6 mm x 6 mm and were screened to eliminate fines using a vibratory apparatus (Retsch AS 200 basic) with a 10 mesh (2.0 mm) sieve. The obtained laboratorial chips had, on average, 8 mm x 3 mm x 2 mm. Heartwood diameters, sapwood radial thickness, and extractives content of these trees were determined and are presented elsewhere (Gominho *et al*. 2015a).

### Methods

*Kraft pulping*

Wood samples collected at the different stem height levels were delignified in a forced circulation reactor with 6 L capacity equipped with an external electric heating system and temperature control. The charge was 1000 g oven dry wood, and the kraft cooking conditions were as follows: 22% active alkali (expressed as $Na_2O$), 30% sulfidity (expressed as $Na_2O$), 4:1 liquor to wood ratio, 70 min time to temperature, and 60 min at the maximum temperature of 160 ºC (H-factor = 460). The pulp was washed with hot water, disintegrated with 2 L of water in a TAPPI standard defibrillator, and screened in a 0.15 mm slot screen (Somerville screener) to remove shives.

The pulps were characterized by the screened pulp yield; the content of shives, the Kappa number (determined by a procedure adapted from TAPPI T236 os-76), and the cellulose polymerization degree was estimated from the intrinsic CED viscosity (SCAN-

CM 15:88). The specific wood consumption (SWC) in $m^3\ t^{-1}$ of oven-dry pulp was calculated using Eq. 1:

$$SWC = 1 / [\text{yield (\%)} * \text{wood density (kg m}^{-3})]*10^5 \quad (1)$$

The wood density values were presented elsewhere (Gominho *et al*. 2015a).

*Bleaching*

The unbleached pulps were bleached using an elemental chlorine free sequence of $D_0ED_1D_2$ (D-chlorine dioxide and E-alkaline extraction). The $D_0$ stage was conducted at 45 ºC, during 30 min, with a chlorine dioxide charge (expressed as active chlorine) corresponding to a Kappa factor of 0.2 (charge % = Kappa number x 0.2). The chlorine dioxide charge (expressed as active chlorine) was 1.3% and 0.6%, respectively in $D_1$ and $D_2$. The $D_1$ stage was carried out at 70 ºC, during 120 min, and the $D_2$ stage was held at 70 ºC, during 180 min, both at medium consistency (10%). The bleaching performance was evaluated by measuring the brightness (ISO 2470-1) and intrinsic viscosity (SCAN-CM 15:88).

*Pulp Characterization*

The bleached pulps were slightly beaten in order to straighten the fibers, at 500 revolutions in a PFI mill and under a refining intensity of 1.77 N $mm^{-1}$ (as defined in ISO 5264-2). The drainability of the pulp suspension (ºSR) was determined by Schopper-Riegler methodology according to ISO 5267-1. The water retention value (WRV) of the pulp fibers was determined by centrifugation of the wet pulp samples during 15 min at 3000 g, according to SCAN-C 62:00. The morphological properties of the pulp fibers, namely the fiber length, width, and coarseness were measured by image analysis of a diluted suspension flowing in a transparent flat chamber observed by a CCD video camera, by measuring more than 8000 fibers using a Morfi® (LB-01) analyzer developed by Techpap (France). Fine elements are defined as particles with size less than 200 µm.

Handsheets were produced with a basis weight of 60 $g.m^{-2}$ and conditioned according to ISO 5269-1 and ISO 187. The properties measured were: bulk density, Bendtsen air permeability, surface roughness, tensile index, tear index, internal bond strength (Scott type, according to TAPPI 569 pm-00), and wet zero-span tensile (according to ISO 5270) and optical properties (brightness and opacity, according to ISO 2470 and 2471, respectively).

## RESULTS AND DISCUSSION

### Raw-Material Characterization

The characteristics of the wood collected at the different heights on the mature *E. globulus* trees are represented in Fig. 1. Heartwood content was high, ranging from 65.9% to 57.7% of the transversal area, respectively, at 0% to 50% of the height level. Heartwood increased with age in proportion of the cross-sectional area and it accumulated more extractives comparatively to sapwood. In commercial trees with 9 to 12 years of age, the heartwood represents on average 40% of the cross-sectional area at the base, and only 10% at the 55% height level (Gominho and Pereira 2000; 2005). Morais and Pereira (2007) observed in trees of 12 to 15 years of age that the heartwood represented 53.3% of the total area at the base and the content of extractives was higher at the base and decreased at the 50% height level (9.8% and 4.2%, respectively). At the usual harvesting age for pulping, extractives content is lower, *e.g.*, 2.9 % (Pereira 1988) or 3.5% (Miranda and Pereira 2001). Heartwood had significantly more extractives than sapwood, with 3.8 and 2.4%, respectively (Morais and Pereira 2012).

The mean basic density decreased upwards from 0.676 g $cm^{-3}$ a base level to 0.652 g $cm^{-3}$ at 50% of the tree height.

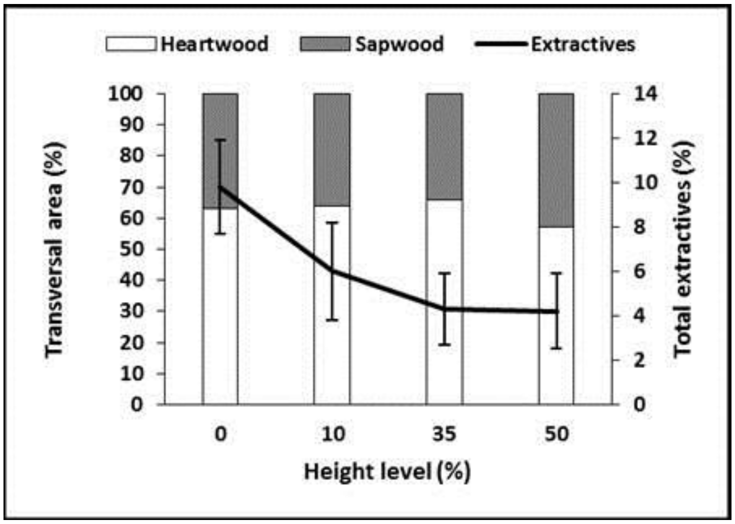


**Fig. 1.** Wood characteristics of mature *E. globulus* wood collected at different height levels: heartwood and sapwood area proportion and extractives content (mean values plus std). Data from Gominho *et al*. (2015a)

## Pulp Properties

Table 1 summarizes the pulp characteristics obtained with the wood collected at different height levels of the mature *E. globulus* trees. The pulping yields increased from 46% to 50%, while Kappa number decreased from 17.5 to 12.3 at 0 to 50% of height, respectively. These differences should be related with the variation of the heartwood proportion and of the extractives content, which decrease from tree base upwards (Fig. 1) since extractives influence negatively the pulp yield (Gutiérrez *et al*. 2001; Lourenço *et al*. 2010). In another study using mature *E. globulus* trees, Gominho *et al*. (2015b) obtained similar pulp yields with a low Kappa number (45% and 11.2, respectively).

The SWC calculated for each level of tree height ranged from 3.2 $m^3$ $t^{-1}$ at 0% height level to 3.1 $m^3$ $t^{-1}$ at 50% height level. Disregarding possible pulp quality issues and the difference in Kappa number, wood samples collected at higher levels will lead to lower pulp costs, since less wood volume will be consumed to make a 1000 kg of pulp (dry matter). In this case, the higher pulp yield at the 50% height level compensates the lower wood density at this level in comparison to the older material at stem base. With 5- to 7-year-old *E. globulus* clones, Guerra *et al*. (2008) found SWC values between 3.7 to 5.0 $m^3$ $t^{-1}$.

The mean pulp intrinsic viscosity was 805 mL $g^{-1}$, decreasing slightly to 732 mL $g^{-1}$ after bleaching. Target ISO brightness was 90% (mean 89.3% ±1.6 for all samples). The intrinsic viscosity of the pulps was low compared to the 942 to 1274 mL.$g^{-1}$ reported by Santos *et al*. (2008), but similar to the 821 mL $g^{-1}$ in Aguayo *et al*. (2012) for *E. globulus* pulps. The mean amount of $ClO_2$ consumed (as active chlorine) was 4.8%, with variations due to the different initial kappa number of the unbleached pulps (Table 1).

**Table 1.** Pulp Characteristics of Mature *E. globulus* Wood Collected at Different Height Levels**

| | | Before bleaching | | After bleaching | | Beating (500 PFI revs) | |
|---|---|---|---|---|---|---|---|
| Height level (%) | Screened Yield (%)* | Kappa number | Viscosity ($mL\ g^{-1}$) | Viscosity ($mL\ g^{-1}$) | Brightness (%) | ºSR | WRV ($g\ g^{-1}$) |
| **0** | 46 ± 1.5 | 17.5 ± 4.1 | 894 ± 114.6 | 805 ± 95.4 | 88.7 ± 1.7 | 20.3 ± 1.9 | 1.07 ± 0.1 |
| **10** | 51 ±1.0 | 15.4 ± 4.1 | 731 ± 45.9 | 707 ± 82.7 | 90.1 ± 2.9 | 18.5 ± 1.0 | 1.00 ± 0.1 |
| **35** | 49 ± 1.2 | 13.3 ± 2.1 | 757 ± 45.1 | 692 ± 37.3 | 89.4 ± 1.0 | 17.0 ± 2.2 | 0.90 ± 0.1 |
| **50** | 50 ± 0.6 | 12.3 ± 1.7 | 838 ± 35.9 | 724 ± 30.8 | 89.0 ± 0.5 | 18.5 ± 1.5 | 0.92 ± 0.1 |
| **Mean** | 49 ± 2.0 | 15.5 ± 3.4 | 805 ± 89.4 | 732 ± 73.3 | 89.3 ± 1.6 | 18.6 ± 1.9 | 0.97 ± 0.1 |

*in all levels the rejects content was less than 0.1%

** mean of 3 trees and standard deviation

The pulps were evaluated after a mild beating aiming at fiber stretching (500 revs in PFI at low beating intensity). The drainability and WRV, used to measure the fiber swelling, changed with the axial position. The pulp from the base presented higher drainability resistance (20.3 ºSR) and WRV (1.07 $g.g^{-1}$) than the corresponding samples from the 50% height level (18.5 ºSR and 0.92 $g.g^{-1}$). These values are in agreement with the pulp fiber morphology described in Table 2.

**Table 2.** Fiber Pulp Morphology of the Unbleached and Bleached Beating Pulps from Mature *E. globulus* Wood Collected at Different Height Levels*

| Height level (%) | Length, weighted in length (µm) | | Width (µm) | | Coarseness ($mg\ m^{-1}$) | |
|---|---|---|---|---|---|---|
| | **UnBle** | **Ble&Beat.** | **UnBle** | **Ble&Beat.** | **UnBle** | **Ble&Beat.** |
| **0** | 827 ± 28 | 829 ± 25 | 19.0 ± 0.6 | 19.5 ± 0.5 | 0.084 ± 0.007 | 0.082 ± 0.003 |
| **10** | 869 ± 12 | 863 ± 13 | 17.9 ± 0.5 | 18.5 ± 0.7 | 0.078 ± 0.003 | 0.081 ± 0.004 |
| **35** | 876 ± 19 | 868 ± 14 | 17.3 ± 0.1 | 17.7 ± 0.2 | 0.074 ± 0.005 | 0.075 ± 0.003 |
| **50** | 877 ± 15 | 874 ± 19 | 17.3 ± 0.4 | 17.8 ± 0.2 | 0.075 ± 0.003 | 0.077 ± 0.001 |
| **Mean** | 862 ± 19 | 858 ± 16 | 17.9 ± 0.4 | 18.4 ± 0.4 | 0.078 ± 0.004 | 0.079 ± 0.003 |
| | **Kinked fibers (%)** | | **Broken ends (%)** | | **Fine elements in area (%)** | |
| | **UnBle** | **Ble&Beat.** | **UnBle** | **Ble&Beat.** | **UnBle** | **Ble&Beat.** |
| **0** | 33.9 ± 0.9 | 29.1 ± 2.6 | 21.6 ± 1.3 | 22.6 ± 1.6 | 9.8 ± 1.4 | 11.5 ± 0.7 |
| **10** | 30.5 ± 1.4 | 26.4 ± 2.6 | 19.7 ± 0.8 | 20.7 ± 1.2 | 7.9 ± 0.4 | 9.7 ± 0.9 |
| **35** | 31.1 ± 1.6 | 27.1 ± 2.5 | 18.6 ± 0.2 | 19.2 ± 0.7 | 7.8 ± 0.3 | 8.7 ± 0.2 |
| **50** | 33.8 ± 2.3 | 27.2 ± 1.0 | 18.1 ± 0.9 | 19.8 ± 0.3 | 7.8 ± 0.5 | 9.0 ± 0.7 |
| **Mean** | 32.3 ± 1.5 | 27.5 ± 2.2 | 19.5 ± 0.8 | 20.6 ± 0.9 | 8.3 ± 0.6 | 9.7 ± 0.6 |

UnBle - Unbleached pulp; Ble&Beat. – Bleached beaten pulp; * Mean of 3 trees and standard deviations

The pulps from the 0% height level presented the lowest fiber length and the highest fiber width which contribute to a more wet state compact structure and therefore, lower resistance drainability (higher ºSR). Both the high WRV (Table 1) and the higher fines content of the pulp at level 0% contribute to the lower drainage rate of the pulps.

The fiber morphology in unbleached pulps shows that fiber length increased (827 µm *vs*. 877 µm) and width decreased (19 µm *vs.* 17 µm), respectively at 0% and 50% height levels. The soft beating process stretched the fibers resulting in a 4% decrease of kinked fibers at all levels. However, the sequential processes of bleaching and soft beating increased the percentage of broken ends and the amount of fines in the pulps.

The pulp fiber morphological features obtained from these mature *E. globulus* trees were slightly different from those obtained from the commercial trees normally used for pulping. For instance, Ferreira *et al*. (2013) reported for *E. globulus* unbleached and unbeaten kraft pulp from a Portuguese mill (Kappa number 16), morphological values of 660 µm length (weighted in length), 16 µm width, 0.087 mg $m^{-1}$ coarseness, and 7.7 % fines content. Using the same equipment as in the present study, Baptista *et al.* (2014) reported 0.070 mg $m^{-1}$ fiber coarseness (using industrial wood chips), fairly lower than the value for mature wood used in the present work. These differences should result from the wood fiber morphology with fiber length and width increasing with age (Jorge *et al.* 2000; Pereira *et al.* 2010).

## Papermaking Properties

The results of the structural, optical, and mechanical properties of the bleached *E. globulus* handsheets are summarized in Table 3. Overall, the characteristics were better when compared with handsheets from commercial *E. globulus* pulp, as referred in the literature (Ferreira *et al*. 2013; Batista *et al.* 2014).

**Table 3.** Structural, Optical, and Mechanical Characteristics of Handsheets Prepared from Bleached Pulps Refined (500 revs. in PFI) from Mature *E. globulus* Wood *

| **Height level** (%) | **Bulk density** ($cm^3$ $g^{-1}$) | **Bendtsen Air Permeability** (ml $min^{-1}$) | **Surface Roughness** (ml $min^{-1}$) | **Opacity** (%) |
|---|---|---|---|---|
| **0** | 1.9 ± 0.1 | 2098 ± 306 | 178.1 ± 25.8 | 82.9 ± 0.8 |
| **10** | 2.1 ± 0.1 | 2442 ± 137 | 217.6 ± 85.2 | 82.1 ± 1.5 |
| **35** | 2.2 ± 0.2 | 2480 ± 128 | 274.7 ± 35.1 | 82.4 ± 1.1 |
| **50** | 2.1 ± 0.2 | 2476 ± 145 | 265.1 ± 60.6 | 82.1 ± 1.1 |
| **Mean** | 2.1 ± 0.1 | 2374 ± 179 | 233.9 ± 51.7 | 82.4 ± 1.1 |
| | | | | |
| **Height level** (%) | **Tensile Index** (N m $g^{-1}$) | **Tear Index** (mN $m^2$ $g^{-1}$) | **Internal bond Strength** (J $m^{-2}$) | **Zero-Span (Wet)** (N m $g^{-1}$) |
| **0** | 37.5 ± 6.2 | 3.4 ± 0.6 | 119.0 ± 16.0 | 109.7 ± 4.8 |
| **10** | 35.0 ± 4.5 | 3.1 ± 0.6 | 92.8 ± 10.9 | 125.5 ± 17.8 |
| **35** | 34.7 ± 5.1 | 3.1 ± 0.5 | 87.5 ± 6.1 | 130.9 ±13.8 |
| **50** | 32.4 ± 5.9 | 2.8 ± 0.9 | 84.0 ± 10.5 | 153.0 ± 39.9 |
| **Mean** | 34.9 ± 5.4 | 3.1 ± 0.6 | 95.8 ± 10.9 | 129.8 ± 19.1 |

*collected at different height levels; mean of 3 trees and standard deviations

Bulk, air permeability, and surface roughness increased from bottom to top in agreement with the decrease of fine elements and the increase in fiber length, which provide a fiber network with increasing porosity. For instance, at 0% height level, the handsheets presented values for bulk, air permeability, and surface roughness, respectively, of 1.9 $cm^3$ $g^{-1}$, 2098 mL $min^{-1}$, and 178.1 mL $min^{-1}$. At 50% height the values were 2.1 $cm^3$ $g^{-1}$, 2476 mL $min^{-1}$, and 265.1 mL $min^{-1}$. When compared with commercial unbleached *E. globulus* pulp (750 revs. in PFI) obtained by Ferreira *et al*. (2013), these mature trees pulps showed higher bulk (mean 2.1 *vs.*1.5 $m^3g^{-1}$) and surface roughness (mean 233.9 *vs.* 167.0 mL $min^{-1}$). The opacity presented similar values along the tree height with a mean value of 82.4%.

The mean values of tensile and tear of 34.9 $Nmg^{-1}$ and 3.1 $mNm^2g^{-1}$, respectively, were lower than the values reported by Batista *et al.* (2014) of 54.8 $Nmg^{-1}$ and 3.46 mN $m^2$ $g^{-1}$ for unbleached kraft pulp produced from industrial wood chips (500 revs in PFI). This is mainly due to the higher densification of the paper structure of the fibers produced from industrial *E. globulus* wood chips (1.51 $cm^3$ $g^{-1}$ *vs.* 2.1 $cm^3$ $g^{-1}$). In fact, in the same work, the handsheet with unbeaten fibers had higher bulk density (1.67 $cm^3$ $g^{-1}$) and the corresponding mechanical properties were consequently lower and more close to those reported in the present paper. Therefore, we can conclude that these mature fibers provided handsheets with good strength properties at higher bulk, which represents an advantage in some paper grades.

The strength properties presented similar trends along the tree: tensile, tear, and internal bond strength decreased upwards while the zero-Span (wet) values increased with height, from 109.7 to 153.0 $Nmg^{-1}$. These variations could be explained by fiber length and fiber curl (Foelkel 2009). This behavior is in agreement with the study made with *E. globulus* overaged sapwood and heartwood where at 500 revs. in PFI the structural, optical, and mechanical characteristics presented similar values, *e.g.*, 2071 and 4325 mL $min^{-1}$ for Bendtsen air permeability, 46.6 and 37.3 N $mg^{-1}$ for tensile index, 2.2 and 1.8 mN $m^2$ $g^{-1}$ for tear index, respectively, for heartwood and sapwood (Gominho *et al*. 2015b).

## CONCLUSIONS

1. The delignification yields of overaged *E. globulus* trees presented yields that increased along the tree height from 46% to 50%, while Kappa number decreased from 17.5 to 12.3;
2. The bleaching response was similar in all levels, but the $ClO_2$ consumption depends on the unbleached kappa number, to achieve 90% of ISO brightness;
3. The morphological properties of the fibers changed with the height level in the trees. The pulp fibers from the base were shorter and coarser than those obtained at 50 % of height. As a consequence, pulp from the base level promoted more kinks and fines during the refining process;
4. The fibers produced from 40-year-old trees provided paper handsheets with better structural, optical, and mechanical properties when compared with handsheets from commercial *E. globulus* usually used by the pulp industry.

## 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: June 9, 2015; Peer review completed: September 26, 2015; Revised version received and accepted: September 30, 2015; Published: October 5, 2015.
DOI: 10.15376/biores.10.4.7808-7816