INCORPORATION OF BARK AND TOPS IN *EUCALYPTUS GLOBULUS* WOOD PULPING

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Bark and the tops of *E. globulus* trees were considered for kraft pulping under industrial conditions. Pulping experiments included wood, bark, tops, and composite samples. Top wood had an average chemical composition most similar to that of wood but with somewhat lower cellulose content (52.8% *vs.* 56.9%) and higher lignin content (18.8% *vs.* 17.8%). There was also a small difference between tops and wood for non-polar extractives, which were higher for tops (2.0% *vs.* 1.4%). Bark had a less favorable chemical composition with more extractives, especially polar extractives (5.3% *vs.* 1.6%) and 1% NaOH solubility (19.9% vs. 12.2%), pentosans (23.7% *vs.* 21.3%), and ash (2.9% *vs.* 1.0%), although the fiber length was higher (1.12 mm *vs.* 0.98 mm). The kraft pulps obtained using bark showed significantly lower yield, delignification degree, and strength properties but had a quicker response to refining. The incorporation of tops and bark in the wood pulping in levels below or similar to a corresponding whole-stem, however, had a limited effect on pulp yield, kappa number, refining, and pulp strength properties. These additional raw-materials, which were estimated to be 26% of the commercial stem wood (14% bark and 12% tops), may therefore be considered in enlarging the eucalypt fiber feedstock in kraft pulping.

Keywords: Eucalyptus globulus; Pulping; Bark; Tops; Whole-tree pulping

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INTRODUCTION

In the pulping sector, the shortage of raw-material is an issue in some regions and is accompanied by rising wood supply costs. Research on alternative or complementary fibre raw materials is well underway, and new tree species, annual plants, and residual materials have been proposed for pulping and paper production (Marrakchi *et al.* 2011; Khiari *et al.* 2010; Patt *et al.* 2006; Khristova *et al.* 2004; 2005; 2006; Gominho *et al.* 2001; Shatalov *et al.* 2001). This is in line with the present framework of using renewable biomass for different purposes combining fibre uses, production of composites, chemicals or biofuels, and energy that can be integrated within the so-called biorefinery concept (Melin and Hurme 2011; Cherubini and Strømman 2011; Carvalheiro *et al.* 2008).

Forest residues are obvious candidates for enhanced use, especially when the residues result from large scale exploitation of plantations. This is the case of *Eucalyptus globulus* Labill., an important species for the production of paper pulp that is grown extensively in different parts of the world, mainly in temperate regions. Portugal was the first country to introduce large scale *E. globulus* plantations and produces high quality bleached eucalypt pulp for printing papers; it remains today a major player in eucalypt pulping with about 740,000 ha of plantations (CELPA 2010). A comprehensive review on the technological quality of *E. globulus* has been published recently (Pereira *et al.* 2010).

The idea of whole tree pulping for an increased use of tree components has been referred to for quite a time, and was a research approach considered in the 70's and 80's of the last century (*e.g.* Laudrie and Berbee 1972; Bublitz 1976; Einspahr *et al.* 1979; Einspahr and Harder 1980; Pereira and Sardinha 1984). A renewed interest arose again in the use of residual or waste biomass materials, namely within biorefinery approaches. In this context barks, branches, tops, and foliage of *E. globulus* and other species have been studied and proposed as sources of valuable materials (Miranda *et al.* 2012a; b).

Eucalypt topwood has a similar composition to the commercial stemwood but with somewhat of a lower cellulose content (58.2% and 50.7% in the stemwood and top, respectively) (Pereira 1988). Bark has more extractives and ash than wood, which are detrimental to pulping (Pereira 1988; Pereira *et al.* 2003) but it also has favourable structural features *i.e.* no rhytidome and a uniform phloem that has a high content of fibers (Quilhó *et al.* 1999; 2000).

This experiment investigated the possibility of using the bark and the tops of *E. globulus* trees for kraft pulping under the same conditions used in the industry. The bark-only and tops-only pulps are presented and compared with wood pulp, while mixed feedstocks of wood with variable amounts of bark and tops were also tested including whole stem experiments. The aim was to provide information for a potential enlargement of the eucalypt fibre supply to the pulp industry, as well as better economics for the eucalypt forest chain.

EXPERIMENTAL PROCEDURE

Sampling

The material came from *Eucalyptus globulus* Labill. trees grown on a commercial plantation located approximately 50 km north of Lisbon, Portugal (Qt. St. António: 39^o 14'N; 9 ^{o} 15'W, 150 m of altitude, air temperature 15.0 ^{o}C, rainfall 745 mm, sunshine 55%, and relative humidity 78%). The plantation used the current silvicultural practices of eucalypt forestry for pulping. The trees were harvested as the first rotation at age 11 years.

Different biomass components within the tree were separated and characterized: wood and bark from the merchantable bole and tops. The merchantable bole was defined as the stem with an over bark diameter greater than 6 cm; tops corresponded to the upper part of the stem and included wood and bark. All the material was chipped before pulping.

The trees biometric data and biomass production by component are shown in Table 1. The average tree wood production in the merchantable bole was 82 kg but considerable variation between individual trees was found. Production of 1 ton of pulpwood will provide approximately 460 kg of forest residues (150 kg of bark, 130 kg of tops, 80 kg of branches, and 104 kg of foliage).

For the experiments, a composite sample of the merchantable bole wood and bark, and the tops was made by mixing the corresponding chips of each tree in their individual mass proportion.

Table 1. Biometric Tree Data (tree height, dbh, and bolewood production in dry matter) and Biomass Component Proportion (in % of bolewood) for the Studied 11-yr-old *Eucalyptus globulus* Trees. Mean of 10 trees, standard deviation, and minimal and maximal values.

Biomass Components	Mean	Std. dev.	Min-Max
Tree height, m	19.5	3.7	13.5 – 26.1
Diameter at 1.3 m, cm	14.8	4	9.5 – 21.6
Bolewood, kg/tree	81.8	60.1	13.9 – 182.7
Bark, % bolewood	15.2	4.3	9.6 – 23.0
Tops, % bolewood	13.5	7.1	3.9 – 24.2
Branches, % bolewood	7.9	2.2	4.3 – 10.9
Foliage, % bolewood	10.2	3.6	4.9 – 16.0

Density

Basic density was calculated by dividing the oven-dry mass by the saturated volume of the sample, determined using the water displacement method,

$$\rho = \frac{W_0}{V_s} \tag{1}$$

where ρ is the basic density (kg/m^3), W_0 is the dry mass (kg), and V_s is the saturated volume (m^3).

Fibre Length

Fibre dimensions were measured on a macerated material using a 1:1 glacial acetic acid with hydrogen peroxide at 40ºC for 48 h for cell dissociation. Two slides with 20 fibres per slide were measured using a Leitz ASM 68 K semi-automatic image analysis system. The cross sectional dimensions of 20 fibres were measured at mid-length. The total diameter and lumen diameter were measured and the cell wall thickness was calculated to be half of the difference between the two diameters.

Chemical Analysis

The chemical composition was determined for each tree biomass component (wood, bark, and top). Sample preparation and determination of ash, extractives, insoluble lignin, pentosans, and 1% sodium hydroxide solubility followed the respective TAPPI standards: ash (TAPPI T211 om-93), non polar and water soluble (TAPPI T 204 om-88, TAPPI T 207 om-93), acid insoluble lignin (TAPPI T 222 om-88), acid soluble lignin (TAPPI UM 250–om-83), 1% NaOH (TAPPI T 212 om-93), and pentosans (TAPPI T 223 cm-84).

The cellulose content was determined by a nitric acidic treatment developed as a modification of the Kürschner and Hoffer (1929) cellulose determination, which has been applied to eucalypt wood (Pereira and Sardinha 1984; Pereira 1988; Wright and Wallis 1998). Extractive-free woodmeal (1 g) was reacted with 25 mL of a nitric acid-acetic acid solution (90 mL of HNO_3 and 732 mL of CH_3COOH made up to 1 liter with water) by boiling for 25 minutes, and the residue was filtered and washed with warm water and ethanol. All chemical determinations were made in duplicates.

Pulping

Kraft pulping was conducted on 400 g (o.d.) samples in a pressurized 7 liter M&K digester with liquor circulation. The pulping was carried out with a liquor-to-wood ratio of 4:1 at 165ºC for 90 min, and the heating time to temperature was set at 30 min. The pulping liquor had a 30% sulfidity and the active alkali was varied between 13% and 22%. Determinations of NaOH and Na_2S in the white liquor were made following TAPPI standard procedures.

The pulping experiments used wood, bark, and tops, as well as composite samples with the following mass proportions: wood and bark (5%, 10%, and 14% of wood); wood and tops (5%, 8%, and 12 % of wood); wood, bark, and tops (14% bark + 12% tops). Inclusion of 14% bark corresponded approximately to the pulping of the unbarked merchantable bole, while inclusion of 14% bark and 12% tops resembled a whole-stem pulping of the *E. globulus* trees.

After pulping, the pulps were washed, disintegrated, and screened through a 0.15 mm slot flat screen and the pulp yield, the screened yield, and the rejects content were determined based on the oven-dry mass of the raw material charged to the reactor.

The unbleached screened pulp samples were characterized by Kappa testing according to TAPPI T 236 om-85.

The black liquors were analyzed for residual alkali by titration with standard hydrochloric acid solution according to TAPPI T-625 cm-85.

Pulp Characterization

The pulps were beaten using a ball mill refiner (Lampen centrifugal mill) with different beating times. The degree of refining was expressed as the drainability, which was measured using a Schopper-Riegler number (ISO 5267-1). The unrefined and the refined pulps were characterized by preparing and testing standard laboratorial handsheets with a grammage of 60 g/m^2 (according to TAPPI T272 om-92) using a Rapid Köthen sheet former. The handsheets were stored at 23 ± 2ºC with 50 ± 2% relative humidity (TAPPI T 402 om-93). The measured physicomechanical properties included: apparent specific gravity, g/cm^3 (TAPPI T 220 om-83), tensile index, Nm/g (TAPPI T 494 om-90), burst index, kPam^2/g (TAPPI T 403 om- 85), and tear index, mN.m^2/g (TAPPI T 414 om-98). Calculations were made to a 43 ºSR freeness level.

RESULTS AND DISCUSSION

Raw Material Characterization

The chemical compositions, basic densities, and fibre lengths of the wood, bark, and tops of *Eucalyptus globulus* are given in Table 2. Overall, the values obtained are within the range reported for the species (Pereira *et al.* 2010).

The basic density of wood and bark is within the range of values reported for *E. globulus* trees at the time of harvest, *i.e.* ranging from 429 kg/m^3 to 600 kg/m^3 for wood, and 374 to 454 kg/m^3 for bark (Quilhó and Pereira, 2001; Ramírez *et al.* 2009). The basic density of bark is significantly lower than that of wood, and therefore when bark is included in the pulping raw material there is a change in the mass-to-volume ratio of the feedstock that decreases the production capacity of the pulping reactors.

Table 2. Fibre Dimensions, Basic Density, and Chemical Composition of Wood, Bark, and Tops of the Studied 11-yr-old *Eucalyptus globulus* Trees. Composite sample of 10 trees.

Fiber and Chemical Characteristics	Wood	Bark	Tops
Density, kg/m^3	602.8	387.6	588.8
Mean fibre length, mm	0.98	1.12	0.91
Fibre width, μm	18.8	18.1	16.2
Wall thickness, μm	4.9	7.0	5.0
Chemical composition,%			
Ash	1.0	2.9	1.0
Non-polar extractives	1.4	1.3	2.0
Polar extractives	1.6	5.3	1.7
Insoluble lignin	17.8	16.9	18.8
Cellulose	56.9	56.0	52.8
Pentosans	21.7	23.7	17.7
1% NaOH solubility	12.2	19.9	14.4

Bark had longer fibers than wood, as reported by Jorge *et al.* (2000), who observed that *E. globulus* bark fibres were approximately 20% longer than wood fibres. Fibre length in the tops was slightly shorter in accordance with Trugilho *et al.* (1996) who found that juvenile eucalypt wood has shorter and thinner fibers.

In regards to chemical composition, the *E. globulus* wood sample showed a high cellulose content and low insoluble lignin content with values similar to those reported in the literature (Miranda and Pereira 2001).

The bark contained more extractives than wood, especially polar extractives (5.3% *vs.* 1.6%) and also had a larger 1% NaOH solubility, pointing out the macromolecular structure differences and the chemistry of cell-wall components differences between wood and bark. Bark was richer than wood in pentosan content (19.9% *vs.* 12.2%). The bark chemical composition values found in this study are similar to what was previously reported by Pereira (1988) for *E. globulus* bark (o.d. wood): total extractives 8.5%, insoluble lignin 21.1%, cellulose 46.7 %, pentosans 18.1%, and 1% NaOH solubility 25.0%. Sakai (2001) also found that *E. globulus* bark as compared to wood contains slightly less lignin, smaller amounts of cellulose, and similar amounts of pentosan.

The chemical composition of tops was comparable to that of wood, but with a somewhat lower cellulose content (52.8% *vs.* 56.9% wood) and slightly higher lignin content (18.8% *vs.* 17.8%). There was also a small difference in non polar extractives between tops and wood, which was higher for tops (2.0% *vs.* 1.4%).

Pulp Yield

The results of the kraft pulping of *E. globulus* wood, bark, and tops and of the wood with different incorporation levels of bark and tops are given in Table 3.

A screened pulp yield of 58.9% with an 18.4 Kappa number was obtained from the wood. Yields decreased for tops and bark, and Kappa number increased: for tops 54.7% yield with a 25.1 Kappa number and for bark 47.2% yield with a 36.1 Kappa number.

In accordance with these results, the inclusion of bark and tops with wood had a negative effect on pulp yield and in delignification levels, although mitigated by the low incorporation levels. The pulping of the unbarked bole (wood with 14 % bark) decreased the pulp yield to 52.0% but the Kappa number increased only marginally. For the option of whole-stem pulping of *E. globulus* (incorporating 14% bark and 12% tops), the pulp yield remained at 52.0% with a small increase in the Kappa number to 22.5.

Table 3. Screened Pulp Yield and Rejects (% of initial dry material), Kappa Number, and Effective Alkali Consumption (%) for the Kraft Pulping at 16% Effective Alkali of Wood, Bark, Tops, and Composite Samples of 11-yr-old *Eucalyptus globulus* Trees

***Eucalyptus globulus* Components**	**Screened Yield (%)**	**Rejects (%)**	**Kappa**	**Effective Alkali Consumption (%)**
Wood	58.9	2.4	18.4	11.99
Tops	54.7	1.9	25.1	11.96
Bark	47.2	2.0	36.1	13.38
Wood + 5% bark	56.8	0.3	18.7	11.16
Wood + 10% bark	53.2	1.6	19.2	11.86
Wood + 14% bark	52.0	2.5	20.2	12.58
Wood + 4% tops	53.5	0.3	16.6	10.48
Wood + 8% tops	53.4	0.4	17.2	10.93
Wood + 12% tops	55.4	1.7	18.1	13.17
Wood + 14% bark + 12% tops	52.0	2.1	22.5	12.65

The loss of pulp yield from using bark in pulping has been often linked to a negative effect of the extractives, *i.e*. high contents of extractives decrease the pulp yields (Amidon 1981). This has been shown repeatedly for *E. globulus* wood with different contents of extractives and specifically also when comparing sapwood to the more extractive-rich heartwood (Gominho *et al.* 2005; Miranda *et al.* 2007). The decrease in yield of 11.2 points when comparing wood and bark pulping (Table 3), however, cannot be assigned exclusively to extractives since the difference in extractives content was only 3.6 points. Another chemical indication of the lower pulp yields of bark is given by a higher 1% NaOH of 7.7 points (Table 3), which indicates a higher solubility of the structural cell wall components.

The anatomical difference between wood and bark certainly plays a significant role in the yield difference due to the loss of the small sized parenchyma cells and phloem vessels in bark during pulping and pulp washing. The parenchyma tissues account for 69% of the volume in the secondary phloem of *E. globulus* (Quilhó *et al.* 2000) and have very thin walls in an anatomic structure that is readily prone to degradation during pulping.

Incorporation of bark had a larger negative effect than the incorporation of tops both in relation to pulp yield and delignification. In fact, the incorporation of tops showed no significant influence (Table 3). This is in agreement with the similar chemical composition of topwood and the same cellular structure as wood with only small cell biometric differences. Jorge *et al.* (2000) noticed only a slight decrease in wood fibre length with an increase in height of *E. globulus* trees.

This whole-tree pulping approach will allow an additional raw-material production per unit area harvested, estimated to be 26% of the commercial stem wood (14% bark and 12% tops), therefore enlarging the eucalypt fiber feedstock for kraft pulping from the commercial plantations, which may be of importance in case of raw-material shortage. The increase in pulp production per unit area harvested will be only 11% given the difference in pulp yield from stemwood and whole-tree pulping (58.9% *vs.* 52.0%).

Pulp Properties

The response to refining differed between the pulps (Fig. 1). Bark pulps were more easily refined and freeness developed faster in bark pulps, *e.g.* to reach the same beating degree of 43ºSR, 27 min of beating was necessary for bark pulp and 52 min for wood pulps (Table 4). The tops pulps were more resistant to beating.

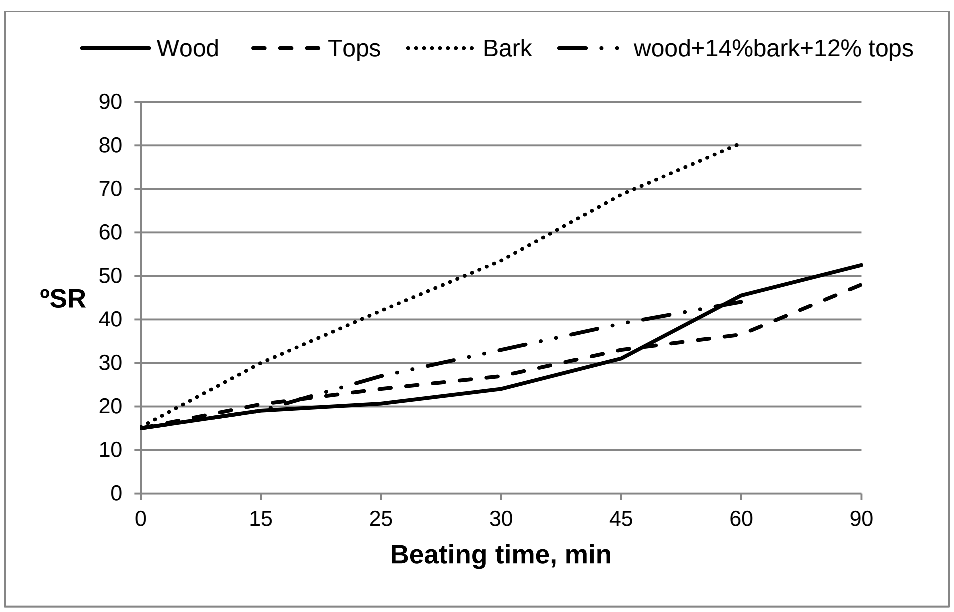

Fig. 1. Freeness development of pulps from wood, tops, and bark during refining

The different refining behavior of wood and bark should be due to the morphological and structural differences. As already referred, bark contains a large proportion of parenchyma and vessels, which are more sensitive to mechanical treatment than wood fibers, degrading fast and creating fines, which increases the beating degree. The higher hemicellulose content of bark may also contribute, since the presence of hemicelluloses is known to increase the swelling of the fibre walls and facilitate the response to refining. Incorporation of tops and bark with wood at the tested levels showed no influence on the refining behavior since the beating times were similar to those of wood only pulps, *e.g.* 54 min *vs.* 52 min of beating.

Refining of pulp fibers is an important factor in the papermaking process, and a determinant for the final paper quality. Consideration of new non-wood fiber raw-materials such as bamboo and straws for pulping therefore requires investigation on their refining ability (Subrahmanyam *et al.* 2000; Gominho *et al.* 2001; Guo *et al.* 2009). The general conclusion is that non-wood fibres respond to refining more easily than wood fibres, as found here in relation to the eucalypt bark.

The results on the bulk and strength properties (tensile, burst, and tear) of the different pulps are summarized in Table 4 for a 43ºSR refining level. The pulp produced from tops was more resistant to tensile and burst, and had the lowest bulk in line with the longer and thinner fibers of the raw material. On the contrary, the bulk of bark pulps was higher, reflective of the more voluminous and less collapsible cellular elements of bark. The paper sheets produced with bark pulp had lower resistance to tensile, tear, and burst compared to wood pulp (Table 4).

The tensile index (78.2 Nm/g), tear index (19.4 mN.m^2/g), and burst index (6.2 kPa m^2/g) of the kraft pulps obtained from wood are comparable to those reported in the literature for the species.

Ramírez *et al.* (2009) reported a tensile index of 90.0 Nm/g, a tear index of 7.8 mNm^2/g, a burst index of 5.6 kPa m^2/g, and a specific bulk of 1.4 cm^3/g for *E. globulus* kraft pulp refined to 30 ºSR. Area *et al.* (2010) reported the following strength properties for *E. grandis* kraft pulps at 30 ºSR: tensile index between 85.2 Nm/g and 103 Nm/g, burst index between 5.3 kPa m^2/g and 7.1 kPa m^2/g, and tear index between 7.8 mNm^2/g and 9.9 mNm^2/g. Mutje *et al.* (2005) reported for a commercial eucalypt kraft pulp at 26.5 ºSR a tensile index of 76.7 Nm/g and a burst index of 7.8 kPa m^2/g.

The strength values are also in agreement with the results of Khristova *et al.* (2006) for kraft pulps from four eucalypts (*E. camaldulensis*, *E. microtheca*, *E. tereticornis,* and *E. citriodora*); tensile index varied between 76.9 Nm/g and 85.0 Nm/g, tear index between 8.4 mNm^2/g and 10.0 mNm^2/g, and burst index between 4.4 kPa m^2/g and 4.9 kPa m^2/g.

Table 4. Pulp Properties for the Kraft Pulping at 16% Effective Alkali of Wood, Bark, Tops, and Wood with Different Incorporation Levels of Bark and Tops for Refined Pulps at 43ºSR

***Eucalyptus globulus* Components**	**Beating time to 43ºSR (min)**	**Bulk (cm^3/g)**	**Tensile index (N.m/g)**	**Tear index (mN.m^2/g)**	**Burst index (kPa.m^2/g)**
Wood	52	1.675	78.2	19.4	6.2
Bark	27	1.963	47.3	13.3	3.6
Tops	84	1.545	83.2	19.1	6.7
Wood + 5% bark	50	1.708	76.0	19.8	6.1
Wood + 14% bark	54	1.616	77.3	18.8	6.4
Wood + 12% tops	58	1.692	77.8	19.2	5.9
Wood + 14% bark + 12% tops	58	1.718	80.9	14.6	5.6

The incorporation of bark and tops had little influence on the corresponding pulp strength characteristics, and the differences were small among the pulps with the several incorporation levels of bark and tops. This shows that the networking of the wood fibres in the paper mat allows the introduction of other fibres and cells without losing the intercellular bonding strength.

On the other hand, some mill operational factors must be considered when incorporating tops and bark in wood pulping: the increased amount of organic material solubilised into the liquor has to be taken into account for black liquor processing in the evaporators and recovery boilers; if there is capacity to process this additional organic load, then more heat will be produced per ton of pulp. The effects on the bleaching process performances as well as environmental issues related to effluents should also be addressed.

At the forest level the potential environmental impact of removing bark and tops should also be considered, namely regarding soil nutrient depletion. However in the usual present forest practice, bark is already removed in a large extent from the commercial plantations (stem debarking occurs at the mill).

CONCLUSIONS

1. In comparison to *E. globulus* commercial stem wood, the topwood presented similar chemical and cellular compositions and may be considered as a possible lignocellolosic source for pulping. The pulp obtained with tops was similar to the wood pulp in regards to yield and strength properties.
2. On the contrary, bark has a less favourable chemical composition with more extractives, pentosans, and ash, although the fiber length is higher. The kraft pulps obtained using bark showed significantly lower yield, delignification degree, and strength properties but were more sensitive to refining.

3. The incorporation of tops and bark in wood pulping at levels below or similar to a corresponding whole-stem had a limited effect on pulp yield, delignification degree, refining, and pulp strength properties. These additional raw-materials, which were estimated to be 26% of the commercial stem wood (14% bark and 12% tops), may therefore be considered in enlarging the eucalypt fiber feedstock in kraft pulping.

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