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AN EXPLORATORY EVALUATION OF THE PULPABILITY OF *BRACHYSTEGIA SPICIFORMIS* AND *PERICOPSIS ANGOLENSIS* FROM THE ANGOLAN *MIOMBO* WOODLANDS

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ABSTRACT

Brachystegia spiciformis and *Pericopsis angolensis* are two hardwood species found in the *Miombo* woodlands. The wood features, kraft pulping and strength pulp properties of both species were evaluated in order to determine their potential as raw material for papermaking. *Brachystegia spiciformis* wood density was 640 kg m^{-3} and *Pericopsis angolensis* was 795 kg m^{-3}. *Pericopsis angolensis* wood has higher cell wall thickness and occluded fibre lumen as remarkable anatomical properties. Runkel ratio, slenderness ratio, and the coefficients of flexibility and rigidity in *Brachystegia spiciformis* were 1.5, 65.7, 41.2% and 29.4%, while in *Pericopsis angolensis* these values were 17.6, 59.9, 5.4% and 47.3%, respectively. *Brachystegia spiciformis* has a higher cellulose content, lower hemicellulose and lignin content, and higher S/G ratio than *Pericopsis angolensis*. In kraft pulping, a higher demand of active alkali was needed for both species, and pulps with high kappa number (24–27) and low pulp yield (40%) were obtained. *Pericopsis angolensis* pulps reached tensile, tear and burst indexes of 99.6 Nm g^{-1}, 5.9 $mN.m^2$ g^{-1} and 4.9 $kPa.m^2$ g^{-1}, respectively. *Brachystegia spiciformis* pulps reached tensile, tear and burst indexes of 100.3 Nm g^{-1}, 10.7 $mN.m^2$ g^{-1} and 6.1 $kPa.m^2$ g^{-1}, respectively. As a conclusion, *Brachystegia spiciformis* wood has better pulpability than *Pericopsis angolensis* wood, according to its pulps properties, despite of the similar pulp yield between both species.

Both species may be suitable for unbleached wrapping papers and rigid cardboards manufacturing.

Keywords: Derived wood properties, kraft pulping, strength properties, wood anatomy, wood chemistry.

INTRODUCTION

Brachystegia spiciformis and *Pericopsis angolensis* are two hardwood species from the *Miombo* woodlands (formation of natural forest, dry and warm predominantly in the South-Central region of Africa). In Angola, this forest occupies an area of 585.949 km^2, which corresponds to 47% of the total country area, being considered mainly as a source for timber production (Figueiredo *et al.* 2009, Sangumbe and Pereira 2014). The wood of *B. spiciformis* is moderately heavy, slightly hard and difficult to be worked, with no significant difference between heartwood and sapwood colours; while *P. angolensis* wood is very heavy and hard, the heartwood is coloured green-brown and the sapwood is grey-yellow (Frost *et al.* 1996, Palgrave 2002). The wood of *B. spiciformis and P. angolensis* are used for construction, cheap furniture, railway sleepers, utensils and beehives. It is suitable for flooring, joinery, mine props, vehicle bodies, crates, veneer and plywood. It is equally important as a source of firewood and charcoal, being among the preferred species for charcoal making throughout Southern Africa (Louppe *et al.* 2008). For another applications of *P. angolensis* wood, Uetimane *et al.* (2009) studied its anatomical properties in order to facilitate the introduction of the specie to the wood industry; Lhate *et al.* (2010) determine the chemical composition and relate it to wood durability and machining properties; while Cuvilas *et al.* (2014) made an evaluation of its wood properties to determine its quality as fuel. Regarding *B. spiciformis* investigations, there are some related species reports available. Atuanya and Ibhadode (2011) evaluated the chemical composition, microstructure and thermal behavior of *Brachystegia nigerica* to determine its potential as reinforcement for polymer

composites. Other studies have reported the yield and strength properties of *B. nigerica* veneer products (Olufemi 2012), evaluations about the ethanol production from *Brachystegia eurycoma* have been made (Afe 2016a, 2016b), and also assessments of the acoustic properties of *B. eurycoma* talking drums (Noah *et al.* 2014).

In Angola, the production of pulp and paper dated back to 1950–1960, where plantations of *Eucalyptus*, *Pinus* and *Cupressus* species were established as fibre source for the national cellulose mill (Companhia de Celulose do Ultramar Português, CCUP) (Silva 1971, Delgado-Matas and Pukkala 2012). After the 1975 independence, 27 years of civil war struck the country, and an extensive illegal logging of the forests occurred due to the lack of fuel sources, in addition to forest fires that destroyed most of the stablished plantations (Delgado-Matas and Pukkala 2012). However, the forestry activities were relaunched in the 2010's, with the beginning of the rehabilitation of the industrial segments related to the pulp and paper production chains (MINADERP 2011). As a consequence, the knowledge of the Angolan *Miombo* species needs to be assessed, as well as, the potential industrial application of wood from these species for chemical conversion processes (pulping, cellulose derivatives, among others). This study is aimed to evaluate the characteristics of the wood, kraft pulping and strength pulp properties of *B. spiciformis* and *P. angolensis* and their potential as raw material for papermaking.

EXPERIMENTAL

Site and sampling

Samples of *Brachystegia spiciformis* Benth and *Pericopsis angolensis* (Baker) Meeuwen were obtained in Sambo, Angola, located at an altitude of 1704 m above sea (13°15'53.61"S and 16°03'52.28"E). The soil is ferrallitic with sandy loam and pH 4.5. The climate is humid with

temperate seasons. The annual rainfall ranging from 1100 to 1400 mm, the average of annual temperature is 20°C and annual relative humidity is 60 to 70%. From one tree of each species (dominant and with straight form) 40 cm logs were taken at 2.5 m from the base of the tree. The age of the specimens was approximately 24 years old for *B. spiciformis* and 40 years old for *P. angolensis.*

Wood material

Logs obtained at 2.5 m of height were cut in 3–4 cm thickness disks. From the disks of each species, 3.0 x 2.5 x 0.2 cm wood chips where handmade for pulping procedures. A fraction of the chips was also milled in a knife mill and sieved to 45/60 mesh for chemical analysis. For wood quality analysis, sapwood and heartwood sections where manually separated from the disks and wood blocks of 2 cm^3 were taken at each section.

Anatomical characterization

Basic density determination

Wood blocks from sapwood and heartwood of each species were used to determine basic density according to the TAPPI Standard Method T258 om-94. Basic density is the ratio of oven dry weight by the green volume, expressed in terms of weight per unit volume. The basic density of wood was calculated according to Heinrichs and Lassen (1970). Basic density measurements of each sample were done in triplicate.

Transversal anatomical characterization

Sapwood and heartwood blocks were macerated with distilled water and glycerine for 7 days. From each block, transversal micro-sections of 30 μm thickness were obtained using a sliding

109 microtome (MICROM, H325). Samples were stained with Safranin and Astra blue, dehydrated
110 with ethanol and assembled in a slide using Canada balsam. Images were obtained using a Zeiss
111 microscope (Primo Star) connected to a personal computer and a digital camera (Canon A640)
112 for image capture. Eighty fibres were randomly selected and their cell wall thickness, fibre width
113 and lumen width were measured with a 100x total magnification. Moreover, eighty vessels
114 randomly selected were measured with a 10x magnification for vessel width and mean vessel
115 area determination. From the captures of 10X magnification the average vessel coverage was also
116 determined, corresponding to the percentage of cross-sectional area covered by vessels. All these
117 parameters were measured using AxioVision Software (Zeiss), which had the proper calibration
118 for each capture lens. Similar procedure was used by Aguayo *et al.* (2010).
119
120 **Fibre quality analysis**
121 Samples of 0.1 x 0.1 x 0.5 cm were obtained from wood chips. Wood samples were macerated in
122 water and treated using Franklin solution (30% hydrogen peroxide and acetic acid, 1:1 v v^{-1}) for 8
123 h at 70°C. The solution was decanted and the remaining fibrous material was washed with water
124 until a neutral pH was achieved. Average fibre length, fibre width, fines content, and coarseness
125 (defined as fibre mass per fibre length) were determined using L&W Fiber Tester equipment
126 (Lorentzen & Wettre, Sweden). L&W Fiber Tester is an instrument for advanced analysis of fibre
127 dimensions. It has a sample feeder where the pulp sample is introduced to the equipment as a
128 suspension for two-dimensional imaging analysis. It consists in two plates that allow the fibres to
129 move freely in two dimensions where a camera captures fibre images for dimension
130 measurements. 200 mg of sample were previously disaggregated in 200 mL of distilled water for
131 10 min. During the analysis of this suspension, the equipment was set to measure approximately
132 35.000 fibres of each sample, setting as fines to elements of 0 to 0.2 mm of length to ensure that

broken fibres and fines are not included in the final averages of fibre measurements. Each sample was analyzed in duplicate.

Derived values of wood fibres

Derived wood properties were calculated from measurements of fibre morphology (Runkel 1949, Luce 1970, Wangaard 1962, Istas *et al.* 1954, Hus *et al.* 1975):

Runkel ratio = (2 x Cell wall thickness) / lumen width

Luce's shape factor = (fibre widht^2 – lumen width^2) / (fibre width^2 + lumen width^2)

Slenderness ratio = fibre length / fibre width

Flexibility coefficient (%) = (lumen width / fibre width) x 100

Rigidity coefficient (%) = (cell wall thickness / fibre width) x 100

Chemical composition of wood

Lignin content

Two grams of milled wood samples were extracted with 90% acetone for 16 h. Samples were characterized for lignin by acid hydrolysis following the methodology described by Mendonça *et al*. (2008). In a test tube were weighed 300 mg of sample and added 3 mL of 72% (w w^{-1}) H_2SO_4. The hydrolysis was carried out in a water bath at 30°C for 1 h with a glass-rod shaking every 10 min. Later, the acid was diluted to 4% (w w^{-1}) with 84 mL of distilled water and the mixture transferred to a 250-mL Erlenmeyer flask and autoclaved for 1 h at 121°C (post-hydrolysis). The residual material was cooled and filtered through a porous glass filter number 3. Solids were dried to constant weight at 105°C and determined as insoluble lignin. Soluble lignin was determined by measuring the absorbance of the solution at 205 nm. Each sample was analyzed in triplicate.

S/G ratio of lignin

The determination of the S/G ratio in the lignin of both species was determined by oxidation with copper (II) oxide carried out following the methodology described by Chen (1992). In an 80-mL stainless steel reactor it was added 400 mg of sample (milled and extracted wood), 2 g CuO, 15 mL NaOH 2 M and 100 mg $Fe(NH_4)_2(SO_4)_2.6H_2O$. Nitrogen was bubbled inside the reactor which was tightly closed and immersed in an oil bath for 3 h at 170°°C. After the reaction, it was added to the reactor 10 mL NaOH 1 M and 10 mL of distilled water. The mixture was acidified to pH 1 with HCl. The content of the reactor was transferred to a centrifuge tube and centrifuged at 2500 rpm for 10 min. The liquid fraction was collected, transferred to a separation funnel and extracted three times with 50 mL of diethyl ether. The organic fraction was further evaporated (40°C) at reduced pressure (450 mbar). The solid residue was dissolved in 1 mL pyridine and 0.5 mL of sample was silylated with 0.5 mL BSTFA. Derivatization products were quantified by GC/FID using the conditions published elsewhere. The amount of syringyl (S) units was the sum of syringaldehyde and syringic acid; and the amount of guaiacyl (G) units was the sum of vanillin and vanillic acid. The amount of S compounds was divided by the amount of G compounds to determine the S/G ratio in lignin. Each sample was analyzed in triplicate.

Cellulose and hemicelluloses determination

The content of cellulose was performed following the Kûrshner-Höffer method, occupied by Carballo *et al.* (2004). On 0.15 g of extractive-free milled wood, 25 mL reactive mixture of HNO_3:C_2H_6O (1:4) was refluxed in a water bath for 1 h, decanted and a new amount of reaction mixture was added, repeating this operation three times, subsequently, 25 mL of 1% KOH was added for 30 min, the residual material was filtered through porous glass filter number 2. Solids

were dried to constant weight at 105°C and determined as wood cellulose. Each sample was analyzed in triplicate. The hemicelluloses were quantified by acid methanolysis according Sundberg *et al.* (1996). Extractive free wood meal was freeze dried prior to weighing 10 mg into a pear-shaped flask. Samples were subjected to acid methanolysis by the addition of 2 mL of 2 M solution of HCl in anhydrous methanol. Samples were kept in an oven at 100°C for 3 h. After cooling to room temperature, 100 µL of pyridine was added to neutralize the acidic solution as well as 4 mL of methanol (containing sorbitol at 0.1 mg/mL as an internal standard). To avoid fibres during silylation, 1 mL of the clear sample solution was transferred into another pear-shaped flask and the solution reduced by rotary evaporation at 40°C. Samples were dissolved in 100 µL pyridine. For silylation, 150 µL hexamethyldisilazane (HMDS) and 80 µL trimethylchlorosilane (TMCS) were added prior to thorough shaking of the sample. After 4 h at room temperature, samples were analysed by GC-FID. One µL of a silylated sample was injected via a split injector (260°C, split ratio 1:20) into a 30 m x 0,25 mm i.d. x 0,25 µm film thickness column DB5. The column temperature program was 100°C to 175°C (4°C min^{-1}) followed by 175°C to 290°C (12°C min^{-1}). The detector (FID) temperature was 290°C. Nitrogen was used as carrier gas. Different peaks were identified by analysing acid methanolysis products of analytical grade sugars (arabinose, xylose, galactose, glucose, mannose, rhamnose, glucuronic acid and galacturonic acid). Calibration curves and factors were determined for each sugar unit in order to calculate the concentration in the samples. Each sample was analyzed in triplicate.

202 **Holocellulose and alpha-cellulose contents**

203 Holocellulose content was determined in extractive-free wood using 250 mg weighed into a 50-
204 mL flask where 5 mL of deionised water, 2 mL of glacial CH_3COOH and 5 mL of 80% $NaClO_2$
205 were added. The flask was closed with a glass cap and was immersed in a water bath at 90°C for
206 1 h. Subsequently, more 2 mL of glacial CH_3COOH and 5 mL of 80% $NaClO_2$ were added to the
207 flask, and the reaction was maintained for 1 h at 90°C. The reaction was quenched by cooling the
208 sample in a water bath at 10°C. The solids were filtered through porous glass filter number 2,
209 washed with 500 mL of deionised water, dried at 105°C until constant weight and determined as
210 holocellulose. To determine the alpha-cellulose content, 100 mg of holocellulose were placed in a
211 25-mL flask, which was treated with 8 mL NaOH 17.5% ($w\ v^{-1}$) for 30 min at room temperature
212 with shaking every 10 min. Then, 8 mL of distilled water was added to the solution and the
213 reaction was carried out for another 30 min. The solids were filtered; the sample was washed with
214 150 mL of distilled water and impregnated with 20 mL of 1 M CH_3COOH for 5 min. The residue
215 was washed with abundant distilled water and dried at 105°C until constant weight for the
216 quantification of alpha-cellulose (Yokoyama *et al.* 2002). Each sample was analyzed in
217 triplicate.

218

219 **Kraft pulping**

220 Kraft pulping was performed in a rotatory digester equipped with four independent 1.5-L reactors
221 (Regmed, Brazil). For each reaction, 100 g of wood chips (dry basis) and cooking liquor with
222 active alkali (AA) concentrations from 14% to 25% and 30% sulfidity (both expressed in NaOH
223 basis) was used. Heating time to the maximum temperature (165°C) was 90 min and the H-factor
224 was 800. The resulting material from each cooking was disintegrated and pulps were screened

through a 0.2 mm slot screen. The pulp was centrifuged to 35% consistency and weighted. The exact moisture was determined and the screened pulp yield was calculated. Kappa number was determined according TAPPI T236 om-99. Strength properties were determined in unrefined and PFI-refined pulps (2500 and 5000 rpm) following TAPPI standard methods for sheet formation (T220 sp-00), tensile index (T404 om-92), tear index (T414 om-98) and burst index (T403 om-97).

Data analysis

Statistical analyses of chemical, anatomical and pulp properties were performed using the software SAS system 9.0 (SAS Institute). Unpaired t-test was used to compare the properties between species.

RESULTS AND DISCUSSION

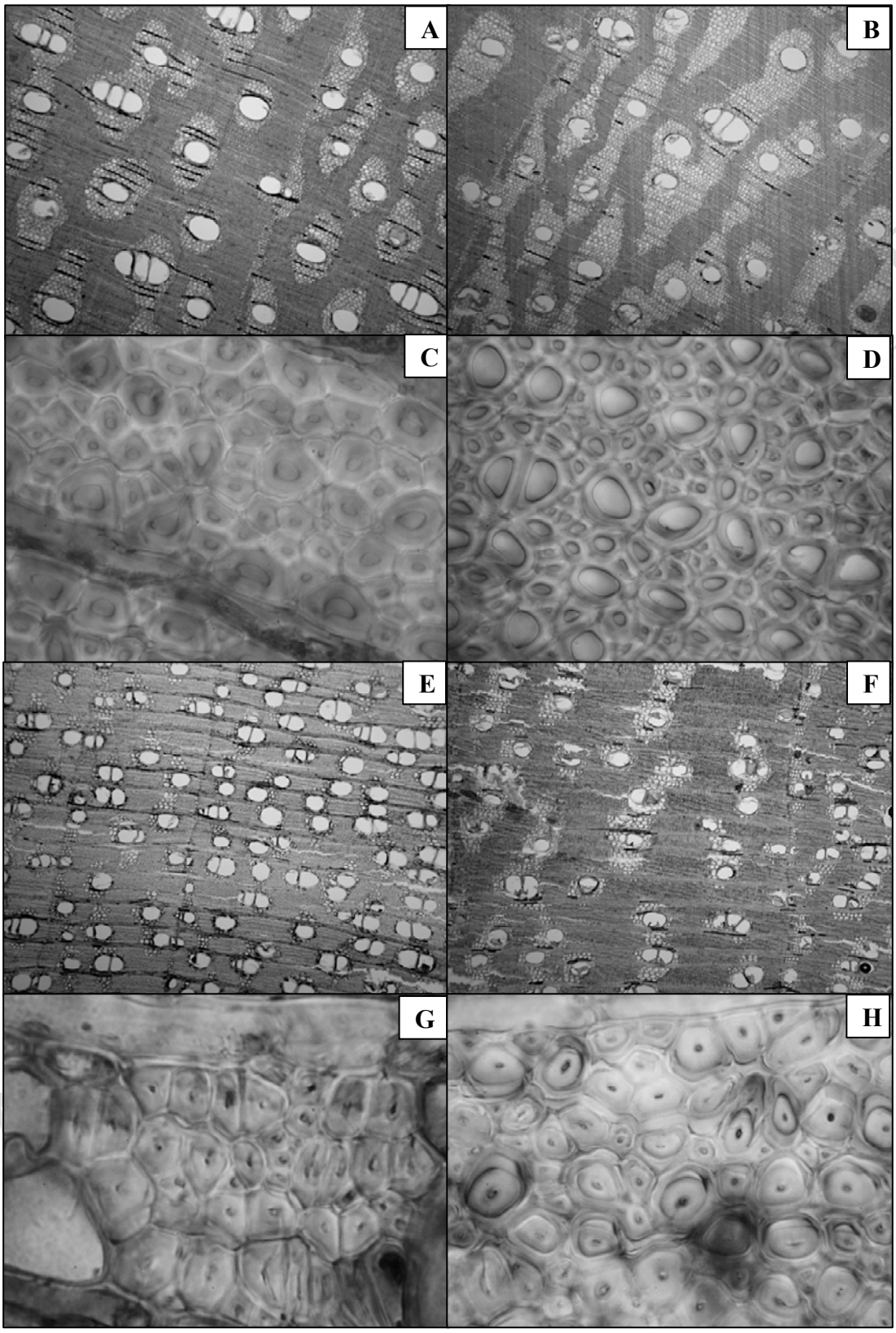
Anatomical characterization of wood

Transversal micrographs showing the cellular anatomy of *B. spiciformis* wood are presented in Figure 1. It can be observed that the vessels are distributed regularly throughout the xylem and are surrounded by aliform axial parenchyma. However, the proportion of parenchyma surrounding the vessel elements is lower in sapwood (Figure 1A) than in heartwood (Figure 1B). It has been showed that the distribution of aliform and paratracheal axial parenchyma arranged as bands gives place to false rings, which also can be associated to the production of fibres with high cell wall thickness (Trouet *et al.* 2001, Grundy 2006). In Figures 1C and 1D, it can be observed the fibre anatomy of sapwood and heartwood, where cell wall thickness is higher in sapwood than in heartwood fibres. It must be also noted that fibre and vessels differences from earlywood and latewood of a growth ring could not be detectable, which is in agreement with the

studies that report difficulties to determine cross-dating by tree ring *Brachystegia* analysis (Trouet *et al.* 2001, Grundy 2006). This is probably due to the ability of the large root system to store water in the dry season (Grundy 2006). Regarding *P. angolensis*, it can be observed a diffuse distribution of vessels in sapwood (Figure 1E) and in heartwood (Figure 1F). However, in heartwood, a higher number of vessels are filled with extractives deposits. Fibre structure of sapwood (Figure 1G) and heartwood (Figure 1H) showed a high cell wall thickness with occluded fibre lumen in both sections. This feature could cause problems for impregnation and diffusion of reagents inside the lignocellulosic matrix during chemical treatments. Similar anatomical features have been reported by Ali *et al.* (2008) and Uetimane *et al.* (2009), who give a more detailed description of the arrangement of xylem cells in *P. angolensis*. Similar to *B. spiciformis* observation, earlywood and latewood in *P. angolensis* could not be detectable.

In Table 1 are presented the features of sapwood and heartwood from *B. spiciformis* and *P. angolensis*. The basic wood density of *B. spiciformis* and *P. angolensis* sapwood was higher than heartwood basic density, while wood basic density average for *B. spiciformis* was 640 kg m^{-3} and for *P. angolensis* was 795 kg m^{-3}. For *P. angolensis*, it has been reported wood density values of 758 kg m^{-3} (Abbot and Lowore 1999), 865 kg m^{-3} (Ali *et al.* 2008), 941 kg m^{-3} (Uetimane *et al.* 2009), 920 kg m^{-3} (Lhate *et al.* 2010) and 865 kg m^{-3} (Cuvilas *et al.* 2014), which is in agreement with our results. Abbot and Lowore (1999) reported for *B. spiciformis* a wood density of 579 kg m^{-3}, including also reports for *Brachystegia boehmii* with 598 kg m^{-3}, *Brachystegia utilis* with 598 kg m^{-3}, *Brachystegia longifolia* with 548 kg m^{-3} and *Brachystegia floribunda* with 676 kg m^{-3}. Also, it was found reports for *B. eurycoma* with wood density of 600 kg m^{-3} (Afe 2016a) and 642 kg m^{-3} (Noah *et al.* 2014). All these findings are in agreement with the density value found in this study for *B. spiciformis*.

Fibre features showed higher values for cell wall thickness in sapwood from both species, being *P. angolensis* fibre cell walls thicker than *B. spiciformis* cell walls (Table 1). Lumen width is also different in heartwood and sapwood from both species, but *P. angolensis* lumen width is occluded as it was already mentioned. Fibre width showed no differences at the different sections of *B. spiciformis*, which is not the same behaviour for *P. angolensis* observations. Regarding vessels features, the area of vessels, vessel coverage and vessel width is higher in sapwood than in heartwood of both species; while *B. spiciformis* showed higher mean area of vessel and higher vessel width average than *P. angolensis*. The radial variation pattern of anatomical properties in hardwoods has been widely report. It is known that the size of vessels increases as cambial age increases (Hudson *et al.* 1998, Leal *et al.* 2003, Carrillo *et al.* 2015). Same pattern of increasing values from pith to bark is known for fibre width, cell wall thickness, fibre length, wood density and coarseness (Miranda and Pereira 2002, Ohshima *et al.* 2004, Quilhó *et al.* 2006, Ramírez *et al.* 2009, Carrillo *et al.* 2015). These assertions are in agreement with the results found for the sapwood section, close to the bark, and the heartwood section, close to the pith, in *B. spiciformis* and *P. angolensis*.



289

290 **Figure 1.** Transversal sections of *B. spiciformis* sapwood at 4X (A), heartwood at 4X (B), sapwood at 100X (C), heartwood at
291 100X (D) and *P. angolensis* sapwood at 4X (E), heartwood at 4X (F), sapwood at 100X (G), heartwood at 100X (H).

292 **Table 1.** Anatomical features of sapwood and heartwood of *B. spiciformis* and *P. angolensis*.

		B. spiciformis		***P. angolensis***	
		Heartwood	Sapwood	Heartwood	Sapwood
Density		630^{b}	650^{a}	740^{b}	850^{a}
(kg m^{-3})	$\bar{x}$	$640^{b} \pm 12$		$795^{a} \pm 64$	
Fibre width		13.68^{a}	13.78^{a}	15.20^{b}	16.80^{a}
(µm)	$\bar{x}$	$13.73^{b} \pm 3.42$		$16.00^{a} \pm 3.25$	
Cell wall thickness		3.71^{b}	4.37^{a}	7.22^{b}	7.91^{a}
(µm)	$\bar{x}$	$4.04^{b} \pm 0.82$		$7.56^{a} \pm 1.54$	
Lumen width		6.37^{a}	4.95^{b}	0.76^{b}	0.98^{a}
(µm)	$\bar{x}$	$5.66^{a} \pm 2.83$		$0.87^{b} \pm 0.49$	
Vessel width		142^{b}	164^{a}	111^{b}	121^{a}
(µm)	$\bar{x}$	$153^{a} \pm 43$		$116^{b} \pm 29$	
Area of vessels		16235^{b}	21179^{a}	10100^{b}	11619^{a}
(µm^{2})	$\bar{x}$	$18707^{a} \pm 7974$		$10860^{b} \pm 5007$	
Vessels coverage		8^{b}	9^{a}	13^{b}	17^{a}
(%)	$\bar{x}$	$9^{b} \pm 3$		$15^{a} \pm 5$	

293 Different letters indicated significant differences between sections of same species and between species (p<0.05).
294

295 **Derived wood properties**

296 Derived values of *B. spiciformis* and *P. angolensis* wood properties are shown in Table 2. These
297 derived wood properties are usually used to predict pulp and paper properties through fibre
298 morphology. In papermaking with hardwood fibres, Runkel ratio lower than 1.0 is desirable for a
299 good paper conformability (Dean 1995, Ona *et al.* 2001, Ohshima *et al.* 2005, Azeez *et al.* 2016)
300 due that fibres are considered as thin walled fibres and good mechanical strength properties are
301 usually obtained (Dutt and Tyagi 2011). Both *B. spiciformis* and *P. angolensis* Runkel ratios are
302 greater than 1.0, which indicates that fibres obtained from this wood species could not be suitable

303 for paper production (Xu *et al.* 2006), however, an index from 1 to 2 have been considered
304 acceptable for papermaking (de Almeida *et al.* 2016).
305
306 Luce's shape factor is related to paper sheet density (Ona *et al.* 2001, Ohshima *et al.* 2005). In
307 14-year-old *E. globulus* trees, values reported were between 0.297–0.329 (Ona *et al.* 2001) and
308 0.390–0.440 (Ohshima *et al.* 2005). However, both *B. spiciformis* and *P. angolensis* Luce's shape
309 factor values are higher than the reported for hardwoods and the reason may be associated with
310 the cell wall thickness, since both fibre width and fibre lumen width are used to obtain the cross-
311 sectional fibre wall area in the equation for Luce's shape factor (Ohshima *et al.* 2005).
312
313 The slenderness ratio is an important parameter related to the physical properties of handsheets
314 such as strength, tear index, burst index and breaking length (Tutus *et al.* 2015). According to
315 those physical properties, the desirable slenderness ratio is between 70–90 in softwoods and 40–
316 60 in hardwoods (Akgul and Tozluoglu 2009). Hence, the ratio obtained in *B. spiciformis* and *P.*
317 *angolensis* was 65.73 and 59.85, respectively. Slenderness ratio in *B. eurycoma* have been
318 reported 71.23 (Olufunmilayo 2013), while in *Eucalyptus* species have been reported between
319 42–66 (Ona *et al.* 2001, Ohshima *et al.* 2005, Dutt and Tyagi 2011).
320
321 Regarding the flexibility coefficient, it has a positive effect on the mechanical strength due to a
322 larger number of bonds between fibres (Dutt and Tyagi 2011). According to Istas *et al.* (1954)
323 there are four types of fibres classified by the flexibility coefficient: (1) High elastic fibres having
324 flexibility coefficient greater than 75%, (2) elastic fibres having a coefficient between 50–75%,
325 (3) rigid fibres having a coefficient between 30–50%, and (4) high rigid fibres with a coefficient
326 less than 30%. *B. spiciformis* fibres can be classified as rigid fibres (41.2%), while *P. angolensis*

showed highly rigid fibres (5.4%). Olufunmilayo (2013) reported for *B. eurycoma* a flexibility coefficient of 63%, while *Eucalyptus* coefficients are between 38 to 74% (Ona *et al.* 2001, Dutt and Tyagi 2011). Concerning rigidity coefficient, *P. angolensis* showed the highest value (47.3%), which influences negatively the tensile, tear and burst of handsheets (Tutus *et al.* 2015).

The fibres with low slenderness ratio, high Runkel ratio and low flexibility are expected to have negative effect on pulp mechanical strength, due that short and thick fibres do not readily collapse to ribbons and provide less surface contact for bonding (Dutt and Tyagi 2011). Stiffer and low flexible fibres form bulky paper of lower bonded area, coarse surfaced and contain a large amount of void volume (Dutt and Tyagi 2011). Therefore, according to the derived values reached, the low elasticity fibres should not be used for writing paper production, but may be used for manufacturing boards, cardboards, rigid cardboards or packaging papers (Dutt and Tyagi 2011, Kiaei *et al.* 2011).

Table 2. Derived wood properties of *B. spiciformis* and *P. angolensis*.

Traits	*B. spiciformis*	*P. angolensis*
Runkel ratio (2 x CWT)/LW	1.5 ± 0.4	17.6 ± 1.9
Luce's shape factor $(FW^2 - LW^2)/(FW^2 + LW^2)$	0.71 ± 0.09	0.99 ± 0.001
Slenderness ratio FL/FW	65.7 ± 0.6	59.9 ± 4.7
Flexibility coefficient (%) (LW/FW) x 100	41.2 ± 7.1	5.4 ± 0.6
Rigidity coefficient (%) (CWT/FW) x 100	29.4 ± 3.5	47.3 ± 0.3

CWT: cell wall thickness, LW: lumen width, FW: fibre width, FL: fibre length.

Chemical composition of wood

The chemical composition of wood from *B. spiciformis* and *P. angolensis* is presented in Table 3. The results showed that existed important differences in the amount of the main components present in both species. The cellulose, holocellulose, alpha-cellulose and acetone-soluble extractives contents, as well as the S/G ratio, were higher in *B. spiciformis* than in *P. angolensis*, while the opposite occurs for hemicelluloses and lignin contents. Interestingly, the S/G ratio of *P. angolensis* is very low (0.89) indicating that the lignin of this hardwood species has higher amount of G-type, instead of S-type units, which would worth a more detailed characterization of lignin in this species in a future work. This is remarkable due that high S-type units promote the delignification and decrease recondensation during the kraft pulping, facilitating the delignification and bleaching processes (del Río *et al.* 2005, Pinto *et al.* 2005, Carrillo *et al.*

2017). Therefore, *B. spiciformis* should show a better performance during alkaline pulping procedures due to its lowest lignin content and highest S-type units amount than *P. angolensis*, in addition to its higher cellulose and alpha-cellulose content. In hardwoods with high pulpability, as *E. globulus* genotypes, the S/G ratio reported is higher (between 2.0–5.5) while lignin and cellulose contents are in agreement with these values (Guerra *et al.* 2008, Aguayo *et al.* 2015).

On the other hand, extractives content affects negatively the pulping procedures, causing increased reagents consumption, inhibition reactions of the delignification process, equipment corrosion and reduction of the pulp quality (Fengel and Wegener 1989, de Almeida *et al.* 2016). In both studied species, the extractive content is acceptable, although in commercial *E. globulus* trees the contents have been reported to be lower (0.5–3.5%) (Martínez *et al.* 2013, Aguayo *et al.* 2014). However, in softwood species the extractives content is higher than 5% (de Almeida *et al.* 2016).

Another noteworthy feature found was for the abundance of some sugars that compose the hemicelluloses. Although the main saccharide found was xylose from the xylans that is a typical hemicellulose found in hardwoods, other minority sugars such as arabinose, mannose, glucose, galactose and rhamnose where significant higher in *P. angolensis,* which could also have a particular polysaccharide structure in wood. During alkaline pulping, the hemicellulose retention plays an important role related with pulp yield and strength properties (Azeez *et al.* 2016), and have been associated to the hemicelluloses structure, molar mass, their substitution degree with methylglucoronic acid and alkali stability in different *Eucalyptus* samples (Martínez *et al.* 2015, Carrillo *et al.* 2017). Consequently, as have been observed in another hardwood species, inherent structural features and content of some particular hemicellulose sugars in *B. spiciformis* and *P.*

389 *angolensis* woods may have an influence on the pulpability of these both species. Atuanya and
390 Ibhadode (2011) reported for *B. nigerica*, 44.5% cellulose content, 20.1% pentosans, 21.2%
391 lignin, 2.4% extractives and 4% ashes, which can be roughly comparable with the values reported
392 in this study. Lhate *et al.* (2010) reported the chemical composition of *P. angolensis* sapwood,
393 outer- and inner-heartwood, with 34% cellulose, 12.7% hemicelluloses, 29.8% lignin and 3.8% of
394 extractives in sapwood. Cuvilas *et al.* (2014) reported a lignin content of 34.7% and extractives
395 of 8.3% in *P. angolensis*.
396
397 **Table 3.** Chemical composition of wood from *B. spiciformis* and *P. angolensis*.

Components (%)	*B. spiciformis*	*P. angolensis*
Cellulose	$50.1^{a} \pm 1.07$	$41.9^{b} \pm 2.12$
Hemicelluloses	$17.6^{b} \pm 0.78$	$26.5^{a} \pm 2.19$
Arabinose	$0.04^{b} \pm 0.07$	$1.04^{a} \pm 0.27$
Mannose	$0.36^{b} \pm 0.08$	$1.24^{a} \pm 0.09$
Xylose	$12.45^{b} \pm 0.05$	$14.58^{a} \pm 0.09$
Glucose	$0.11^{b} \pm 0.07$	$3.38^{a} \pm 0.35$
Galactose	$0.09^{b} \pm 0.07$	$1.37^{a} \pm 0.14$
Rhamnose	$0.04^{b} \pm 0.08$	$1.28^{a} \pm 0.28$
Uronic groups	$4.52^{a} \pm 0.64$	$3.58^{b} \pm 1.06$
Acetone/water extractives	$5.6^{a} \pm 0.24$	$4.6^{a} \pm 0.95$
Lignin	$22.5^{b} \pm 0.7$	$29.2^{a} \pm 0.22$
Holocellulose	$68.0^{a} \pm 0.37$	$67.7^{a} \pm 1.09$
Alpha-cellulose	$49.3^{a} \pm 1.8$	$40.0^{b} \pm 1.58$
Lignin S/G ratio	$1.72^{a} \pm 0.07$	$0.89^{b} \pm 0.08$

398 Different letters indicate significant differences between species ($p<0.05$).

Kraft pulping

Kraft pulping of wood chips from *B. spiciformis* and *P. angolensis* was performed at an H-factor of 800 and different active alkali charges (14% to 25%) with the aim to obtain bleachable-grade pulps (Figure 2A). For the most common hardwood species used to produce bleached pulps, such as *Eucalyptus*, acacia or birch, active alkali charges of 16%–24% are usually enough to generate bleachable-grade pulps with kappa number around 16–19 and pulp yield of 50-55% (Pinto *et al.* 2005, Aguayo *et al.* 2010). In the present case, for both species, the demand of active alkali was higher (up to 25%) and pulps with high kappa number (24 for *B. spiciformis* and 27 for *P. angolensis*) were obtained, at the cost of a lower screened pulp yield (approximately 40%) for both species (Figure 2B). *P. angolensis* was harder to pulping than *B. spiciformis* since it presented higher kappa number during cooking at different alkali charges, associated with lower pulping yield (Figures 2A and 2B, respectively). The main factors that affect the lower pulpability of *P. angolensis* could be related with the anatomical features. High-density woods (more than 600 kg m^{-3}) are usually associated with tylosis formation or extractives deposition in lumens that lead to an irregular delignification of wood chips. Which is mainly due to the poor efficiency of the impregnation with the cooking liquors (Ramírez *et al.* 2009). Anatomically, *P. angolensis* wood also presented occluded fibre lumen, which can represent an important drawback for reagent diffusion during pulping procedures. Chemical features such as, extractives amount, lignin content and S/G ratio are also known to affect the delignification rate and alkali consumption. As already mentioned, *P. angolensis* showed the higher lignin content and higher G-type units (Table 2). G-type lignin contains more resistant linkages than S-type units, making lignin less reactive and resistant to chemical degradation (Boerjan *et al.* 2003, Pinto *et al.* 2005, Del Río *et al.* 2005, Rencoret *et al.* 2008).

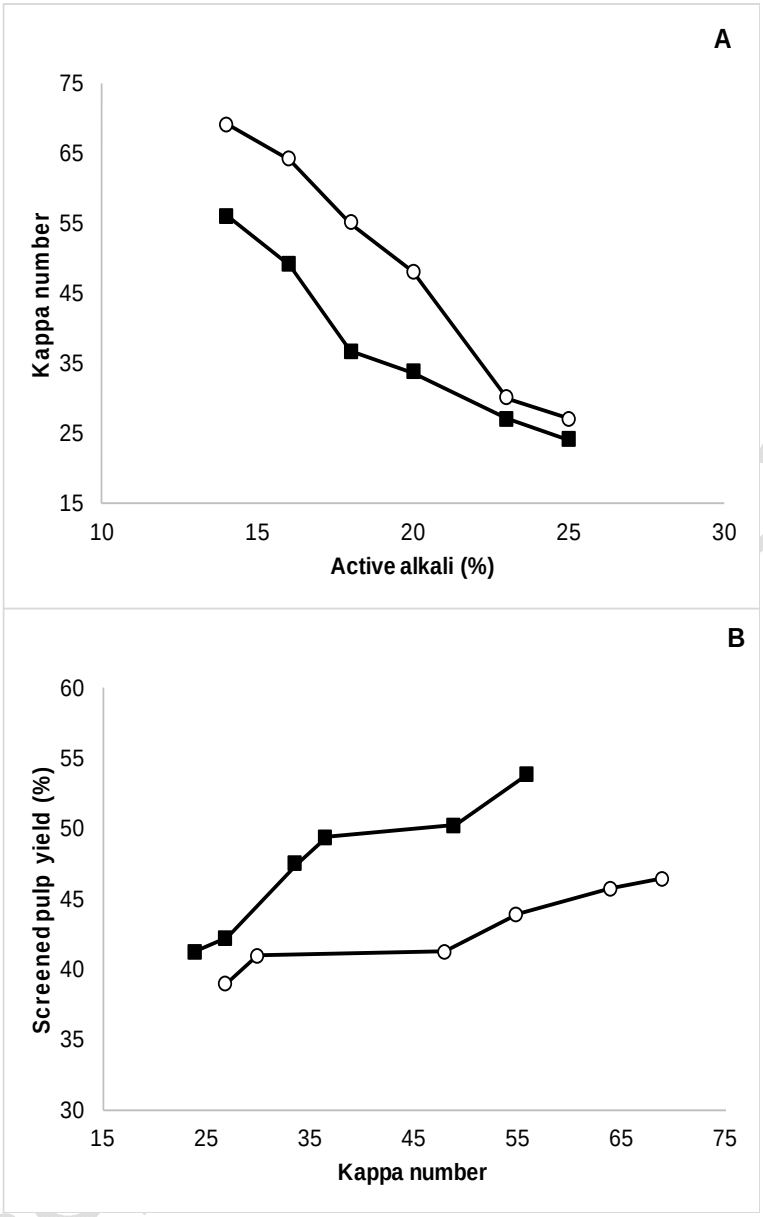
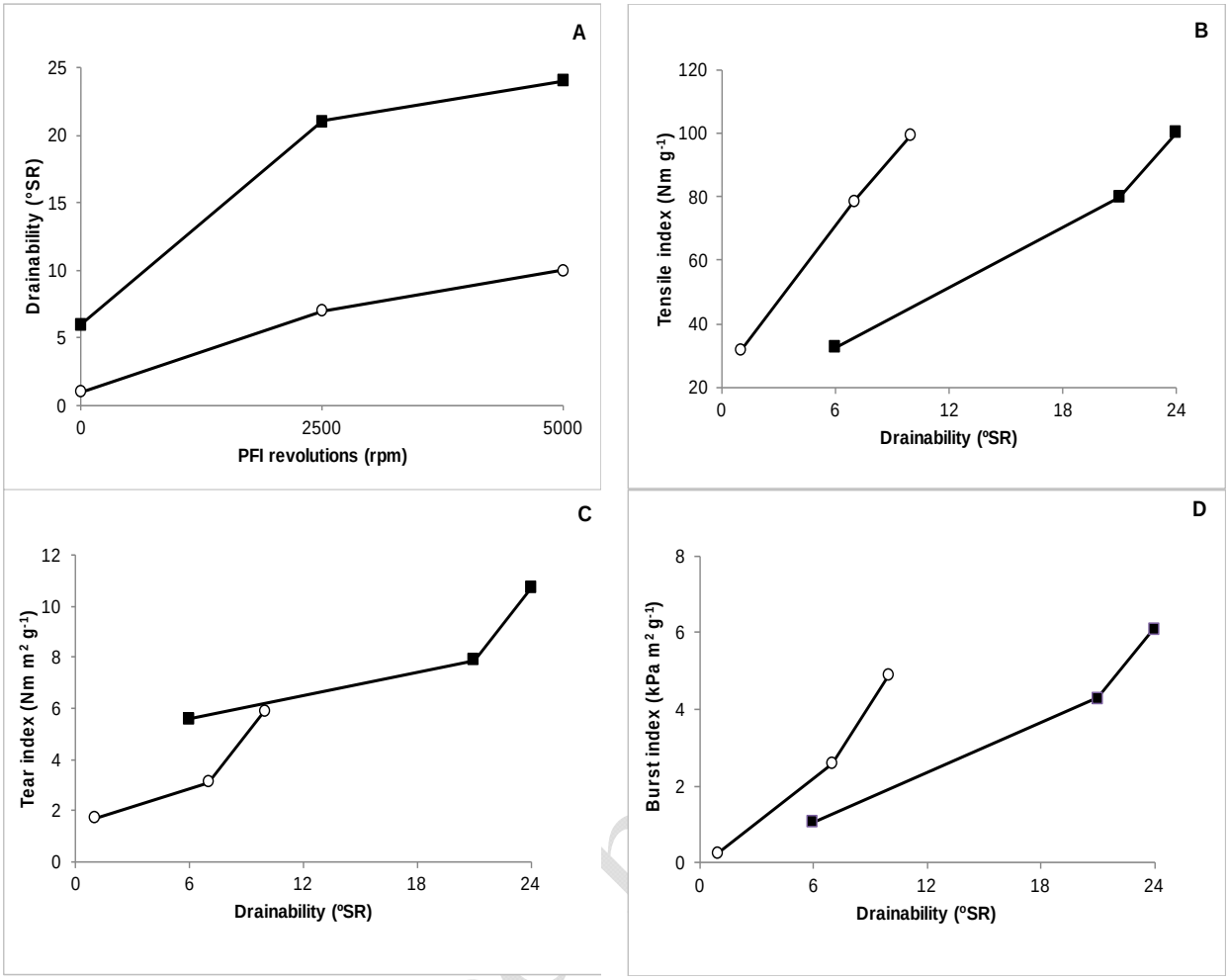


Figure 2. (A) Kappa number and (B) delignification selectivity after kraft pulping of *B. spiciformis* (-■-) and *P. angolensis* (-❍).

Unbleached kraft pulps were refined at 2500 and 5000 revolutions in a PFI mill to increase internal and external fibrillation of pulp (Figure 3A). Only *B. spiciformis* was able to be refined at values higher than 20°SR (usually required for high strength papers) achieving 24°SR at 5000 rpm. *P. angolensis* was harder to refine and only poor fibrillation was obtained at 5000 rpm (10°SR). The main reason of the poor fibrillation of *P. angolensis* may be related with the

morphological features of its fibres. As was mentioned, its highest cell wall thickness and occluded lumen contribute to the decreasing of fibres elasticity (Runkel ratio of 17.6 and flexibility coefficient of 5.4%), demonstrating that stiff fibres are harder to refine and process. Figure 3B, C and D shows the strength properties of *B. spiciformis* and *P. angolensis* pulps. Tensile, tear and burst indexes of 100.3 Nm g^{-1}, 10.7 mN.m^2 g^{-1} and 6.1 kPa.m^2 g^{-1}, respectively, were reached for *B. spiciformis* pulps after refining at 5000 revolutions. The pulps of *P. angolensis* after refining at 5000 revolutions reached tensile, tear and burst indexes of 99.6 Nm g^{-1}, 5.9 mN.m^2 g^{-1} and 4.9 kPa.m^2 g^{-1}, respectively. As was already discussed, the lowest strength properties of *P. angolensis* were expected, mainly due to the influence of the low slenderness ratio and flexibility coefficients, and high Runkel ratio of its fibres (Table 2). As a reference of strength properties in commercial hardwoods, Guerra *et al.* (2008) reported for kraft *E. globulus* pulps tensile indexes of 66–102 Nm g^{-1}, tear indexes of 6–9 mN.m^2 g^{-1} and burst indexes 5–8 kPa.m^2 g^{-1}.



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453
454
455 **Figure 3.** (A) Drainabiity and pulp strength properties of *B. spiciformis* (-■-) and *P. angolensis* (-
456 ❍). (B) Tensile index, (C) tear index and (D) burst index.
457

458 In Table 4 are shown some biometric properties of pulp fibres after the refining process as fibre
459 length, coarseness, fines content and kink index. Fibre length is an important descriptor factor of
460 pulp quality, due to its influence to paper strength properties (Ek *et al.* 2009). Fines content is
461 related with amount of short and thin cells as parenchyma cells and broken fibres, and have been
462 reported that it influences positively the sheet tensile index (Retulainen 1997). The kink index
463 refers to a deformation on the fibre that can be a weak or a breaking point; while coarseness,
464 defined as fibre mass per fibre length, is a good index for predicting pulp properties of fibres and
465 basic density of wood (Via *et al.* 2004, Mansfield and Weineisen 2007, Carrillo *et al.* 2015). The

466 fibre length and coarseness of both species decreases after refining, while kink index and fines
467 content increases. These results are expected considering that during refining some fibres are
468 more likely to bend and break, increasing the fines release and kink index of pulp fibres.
469 Particularly, *P. angolensis* pulps showed the higher values for fibre length, fines content and
470 coarseness, and lower values for kink index during the different refining steps, which is probably
471 due to its higher fibre cell wall thickness. However, despite the physical features of pulp fibres,
472 *B. spiciformis* showed better strength pulps properties than *P. angolensis*, which could be
473 considered unexpected according to pulp fibres features. Nevertheless, very-high coarseness and
474 very-high cell wall thickness values can lead to poor conformability and low fibre-to-fibre
475 contact in sheets (Dean 1995), which resulted in poor derived wood properties and low strength
476 properties as in *P. angolensis*.
477

478 **Table 4.** Fibre biometry of *B. spiciformis* and *P. angolensis* pulps.

	B. spiciformis			***P. angolensis***		
	PFI revolutions (rpm)			PFI revolutions (rpm)		
	0	2500	5000	0	2500	5000
Fibre length (mm)	1.02[a] ± 0.01	0.93[b] ± 0.01	0.92[b] ± 0.01	0.98[a] ± 0.01	0.97[a] ± 0.01	0.95[b] ± 0.01
Coarseness (mg/100 m)	10.3[a] ± 1.1	8.4[b] ± 1.7	6.9[c] ± 0.1	11.5[a] ± 0.3	11.2[a] ± 2.3	10.6[b] ± 0.9
Kink index	0.90[b] ± 0.03	1.92[a] ± 0.01	2.40[a] ± 0.42	0.58[b] ± 0.08	0.72[b] ± 0.03	1.34[a] ± 0.03
Fines content (%)	2.9[b] ± 1.05	3.2[b] ± 0.01	5.7[a] ± 0.07	2.5[c] ± 0.07	6.7[b] ± 0.21	8.3[a] ± 0.28

479 Different letters indicate significant differences between refined pulps of the same specie (p<0.05).
480

CONCLUSIONS

P. angolensis and *B. spiciformis* are species acknowledged by their heavy and hard wood properties. However, *P. angolensis* wood has higher wood density, higher fibre width and cell wall thickness, and an occluded fibre lumen as remarkable anatomical properties. Regarding chemical features, *B. spiciformis* has a higher cellulose content, lower hemicellulose and lignin content, and higher S/G ratio than *P. angolensis*. The kraft pulping procedure of both species required higher alkali charges to achieve bleaching grade pulps, resulting in low pulp yield and high kappa number. The pulps obtained from *P. angolensis* wood were harder to refine and showed lower strength properties than *B. spiciformis*, which was attributed to the very-high values obtained for biometric properties and not adequate derived wood properties of wood and pulp fibres of *P. angolensis*, as wood density, cell wall thickness, Runkel ratio, slenderness ratio, flexibility coefficient and coarseness. It is concluded that *B. spiciformis* wood has better pulpability than *P. angolensis* wood, according to its pulps properties, despite of the similar pulp yield between both species. *B. spiciformis* and *P. angolensis* may be not suitable for high quality papers, but both species could be useful for unbleached wrapping paper and rigid cardboards.

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