Characterization of Cocoa (*Theobroma cacao* L.) Wood Branches as a Potential Resource for Paper Production

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For sustainable use of lignocellulosic resources, pruned tree branches of cocoa, one of the major evergreen tropical tree crops with significant economic importance worldwide, were investigated as a potential source of kraft pulp. This study determined the chemical composition, fiber dimensions, kraft pulp, and paper properties of the cocoa tree (*Theobroma cacao* L.) branches compared to the deciduous trees traditionally used in the paper industry. A handsheet of cocoa pulp showed promising results with narrow fiber length distribution, high paper density, and high mechanical strength. The yield of kraft pulp made from cocoa branches wood was lower. The tensile and burst indices of cocoa pulp handsheet were 2 and 2.5 times higher than that of hardwood traditionally used in the paper industry. These results suggest a potential use of cocoa (*Theobroma cacao* L.) tree branch wood for pulp production using the kraft process.

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INTRODUCTION

To sustainably use the available lignocellulosic resources, the biomass of any plant, especially wood-like, should be evaluated as a potential raw material for bio-based materials. Wood branches are recognized as a considerable carbon source, not far behind the stem (Viera and Rodríguez-Soalleiro 2019). They have been extensively studied as a cellulose fiber source or for other applications. Various fruit trees or other trees whose primary use is not timber production have been investigated, *e.g.*, walnut tree branches (Guo *et al.* 2022), root and branch wood of *Populus ussuriensis* Kom. (Zhao *et al.* 2018), branch wood of *Acacia gerrardii*, *Tamarix aphylla*, and *Eucalyptus camaldulensis* (Suansa and Al-Mefarrej 2020), *Betula costata* Trautv (Zhao *et al.* 2019b), deciduous branches and softwood spindles (Olarescu *et al.* 2022), branches of *Betula platyphylla* Roth. (Zhao *et al.* 2019a), and *Pinus halepensis* and *Eucalyptus camaldulensis* grown in Egypt (Hassan *et al.* 2020). Branch wood of two *Tilia* species has been recognized as a potential source for high pulp yield (Guo *et al.* 2023), and wood fibers from discarded parts of pomelo trees (Hung *et al.* 2021) have also been investigated. White oak branches have been studied as a source of wood flour for reinforcement of polypropylene materials (Hernández-Jiménez *et al.*

2022), while pruned apple tree branches and pruned *Eucommia ulmoides* branches have been used for the production of vinegar for different applications (Liu *et al*. 2021; Xue *et al*. 2022). Insulation boards have been made of camphor branches (*Cinnamomum camphora*) (Cai *et al*. 2024).

Cocoa (*Theobroma cacao* L.) is a major evergreen tropical tree crop because of its economic importance worldwide. Cocoa trees grow or are grown in areas close to the equator (20 to 25 degrees north and south in latitude), mostly in Africa and Central and South America. A cocoa tree can reach up to 20 m in the wild, but it is preferable to maintain a maximum of 6 to 8 m plants for effective management. The main producing countries are Cote D'Ivoire, Ghana, Indonesia, Ecuador, and Cameroon, with a total contribution of over 70% (Mendoza-Meneses *et al.* 2023). The demand for cocoa products has been increasing by an average of three percent per year since 1999, and further growth is expected to satisfy the growing global demand driven by emerging economies (Fairtrade Foundation 2016).

Pruning is a common agricultural and horticultural technique primarily used to alter the structure of trees. This practice helps control tree size, enhance light capture efficiency, boost yield and fruit quality, and simplify harvesting. Furthermore, pruning is acknowledged as one of the most necessary tools to control the total productivity of cocoa agroecosystems (Mendoza-Meneses *et al.* 2023). Different types of pruning are performed on cocoa trees: formation pruning to create the structure of the crown after the tree has developed the first whorl of branches, and structural and maintenance pruning, which are performed one or several times per year to reduce excessive self-shading and maintain correct tree dimensions (Tosto *et al.* 2023). This pruning becomes a significant source of wood biomass, which usually remains on the plantation or is used as firewood. Collecting branch wood has been recognized as a significant factor in increasing the quantity of wood fiber per forest area for species traditionally used in the pulping industry. However, branch wood limitations need to be taken into account, such as lower quality of fibers because of anatomical features typical for branch cells (Suansa and Al-Mefarrej 2020).

The properties of the natural fiber materials are determined by each fiber's physical, chemical, morphological, mechanical, and other parameters. Recently, cocoa bean shell, cocoa tree fruit residue, was evaluated as a precursor for cellulose nanofibrils using a mechanical defibrillation grinder (Souza *et al.* 2019), and paper packaging material has been developed from fibers of cocoa shell (James Copper Paper 2013). However, to the best of the authors' knowledge, there is a lack of research on cocoa branch wood pulp fibers and their properties. Therefore, the present study aims to evaluate the potential of cocoa branch wood as a source of cellulose fibers and to characterize these fibers in terms of their suitability for papermaking applications. The study hypothesizes that cocoa branch wood is a suitable feedstock for kraft pulping, and the resulting fibers possess adequate properties for papermaking.

EXPERIMENTAL

Materials

Branches (2 to 6 cm in diameter, 10 to 15 cm in length) obtained during the pruning process of the cocoa tree (*Theobroma cacao* L.) were collected from "Centro de investigacion La Suiza de Agrosavia" (Santander, Colombia) in March 2022. The bark was manually peeled off the branches; branch wood was dried at room temperature and stored dry until used (Fig.1). Debarked birch (*Betula pendula*) wood was supplied by the A/S Latvijas Finieris company "Lignums" (Riga, Latvia) and was analyzed as the reference wood often used in traditional Nordic pulp and paper industry.

Fig. 1. Cocoa branch wood samples, debarked and dried

Methods

Fourier transform infrared spectroscopy

Fourier transform infrared (FTIR) spectra of cocoa branch wood and birch wood were recorded in KBr (IR grade, Sigma Aldrich, Darmstadt, Germany) pellets using a Thermo Fisher Nicolet iS50 spectrometer (Waltham, MA, USA) in the range of 4000 to 450 cm^{-1} with a spectral resolution of 4 cm^{-1} and number of scans of 32. The pellet contained approximately 2 mg of the ball mill (MM200, Retsch, Germany) ground wood sample and 200 mg KBr. Both spectra were normalized to the highest absorbance maxima.

Gravimetric chemical characterization

Samples were ground in a Kika Werke M20 mill (Kika GmbH&Co, Staufen, Germany) to pass through a 0.6-mm screen. The grounded samples were extracted with acetone (Sigma Aldrich, Germany) for 8 h to quantify extractable components according to the TAPPI T280 pm-99 standard (2000). The content of Klason lignin was determined according to the TAPPI T222 om-11 standard (2021). The ash content in wood was analyzed according to the TAPPI T211 om-22 (2015).

Chemical composition analysis

Wood sample preparation for high-performance liquid chromatography (HPLC) analysis was performed according to Sluiter *et al.* (2008). The HPLC analysis of the obtained hydrolysates was performed to determine the content of monosaccharides, 2-furaldehyde, 5-hydroxymethylfurfural (5-HMF), and organic acids using a Shimadzu LC-20A HPLC (Shimadzu, Tokyo, Japan) with a refractive index detector. Glucose, xylose, cellobiose, arabinose, galactose, mannose, 2-furaldehyde, acetic acid, 5-HMF, levulinic acid, and formic acid (Sigma Aldrich, Germany) with purity ≥ 99.0% were used as reference standards. Glucose, cellobiose, 2-furaldehyde, acetic acid, 5-HMF, levulinic acid, and formic acid were analyzed on a Shodex Sugar SH1821 column at 60 °C, with eluent 0.008 M H_2SO_4 (Sigma Aldrich, Germany) at a flow rate of 0.6 mL•min^{-1}. Carbohydrates were determined on the Shodex Sugar SP0810 column at 80 °C, with deionized water as the mobile phase under a flow rate of 0.6 mL•min^{-1}. $NaHCO_3$ was used to neutralize the sample from pH 5 to 7. Samples were filtered through a 0.45-µm membrane filter before injection.

Kraft cooking

Cocoa branch wood and birch wood were chipped manually. NaOH and Na_2SO_4 with purity ≥ 99.0% were purchased from Sigma Aldrich, Germany. Kraft pulping was performed as a one-stage delignification in a custom-made semi-automatic 2-L laboratory digester (SIA "Fil un Ko," Latvia). Wood chips were put in the digester, and white liquor (57.4 g/L active alkali and 29.8% sulphidity) was poured over the wood with liquor wood ratio of 4.5:1 (Sable *et al.* 2013). The temperature of the digester reached 165°C in 30 minutes, and then cooking was performed for 85 minutes. After cooking, the digester was cooled with cold water until room temperature. The kraft pulp was rinsed with tap water until a clear filtrate with pH 7 was obtained. The pulp was dried at room temperature and stored dry until used. Figure 2 shows the initial cocoa branch wood sample and the dry pulp obtained. The yield was calculated by dividing the pulp oven-dried mass by the initial wood oven-dried mass and expressed as a percentage.

Fig. 2. Cocoa branch wood sample and obtained unbleached kraft pulp sample

Bleaching

Bleaching was performed on the 4% fiber suspension using H_2O_2 and NaOH at 5% and 1.5% relative to the oven-dried fiber mass. To assess and compare the bleachability of investigated pulps under light conditions, a simple one-stage bleaching method was selected from the methods usually used in the bleaching sequence (Treimanis *et al.* 2009). H_2O_2 solution (36% assay) was purchased from Sigma Aldrich, Germany. The bleaching process was carried out for 90 min at 80 °C. After bleaching, the fibers were filtered, washed with deionized water until neutral pH, and dried for storage until further use.

Brightness

Unbleached and bleached paper handsheet brightness was measured according to standard ISO 2470-1:2016 using the spectrophotometer Elrepho (Lorentzen & Wettre, Kista, Sweden). The diffuse blue reflectance factor was measured as parameter R457 D65 from the list provided by the spectrophotometer.

Scanning electron microscopy (SEM)

Samples were prepared for SEM imaging by applying a thin layer of gold plasma using a K550X sputter coater (Emitech, South Petherton, UK). Subsequent examination was conducted using a Vega TX microscope (Tescan, Czech Republic), running software version 2.9.9.21. Images were taken at 200x and 300x magnification.

Fiber and handsheet properties

Fiber properties (length, width, and shape) were determined with the "FiberTester" (Lorentzen & Wettre, Kista, Sweden). Standard paper handsheets were prepared according to ISO 5269-2 (2004) with a Rapid Köthen paper machine (Frank PTI, Birkenau, Germany) and conditioned following ISO 187 (2022). Handsheets of the unbeaten pulp were prepared at the standard grammage 75 g/m^2 determined by ISO 536 (2019). Tensile and burst strength was measured according to ISO 1924-2 (2008) and ISO 2758 (2014), using a FRANK Tensile Tester Vertical F81838 and FRANK Burst Tester for Paper (Frank PTI, Birkenau, Germany), respectively. Determination of the Schopper-Riegler degree was performed as per ISO 5267-1 (1999), determination of grammage as per ISO 536 (2019), and thickness, density, and specific volume (bulk) as per ISO 534 (2005).

Statistical analysis

The statistical analysis was performed using an analysis of variance (ANOVA) Single Factor and Correlation by Excel data analysis (Microsoft Corp., Redmond, WA, USA). All statistical tests were conducted at a significance level of $\alpha = 0.05$.

RESULTS AND DISCUSSION

The FTIR spectroscopy provided general information about the chemical composition of the cocoa branch wood sample. The spectrum of birch wood was used as a reference sample (Fig. 3). Both spectra were normalized to the highest absorption maxima. The spectra showed almost a similar absorbance maxima position with some intensity differences. The absorbance bands used for analyses were assigned based on established references (Bellamy 1980; Faix 1992): a broad peak at 3420 cm^{-1} for OH groups, around 2919 cm^{-1} for aliphatic (-CH_2-, -CH_3) stretch region, 1739 cm^{-1} for unconjugated C=O of the aliphatic ester groups (mainly, acetyl groups), peaks at 1596, 1507, and 1424 cm^{-1} mainly for aromatic skeletal vibrations of lignin, peak at 1463 cm^{-1} for asymmetric C-H deformation in -CH_3 typical for lignin methoxyl groups, and a peak at 832 cm^{-1} for C-H out of plane vibration of 2 and 6 position of lignin syringyl ring. The highest absorbance maxima at the fingerprint region of both spectra corresponded to C-O, C-O-C, and C-O-H vibrations around 1050 cm^{-1}, typical for sugars. Based on the 1050/1507 cm^{-1} absorbance ratio, the cocoa branch wood contained more lignin than birch wood.

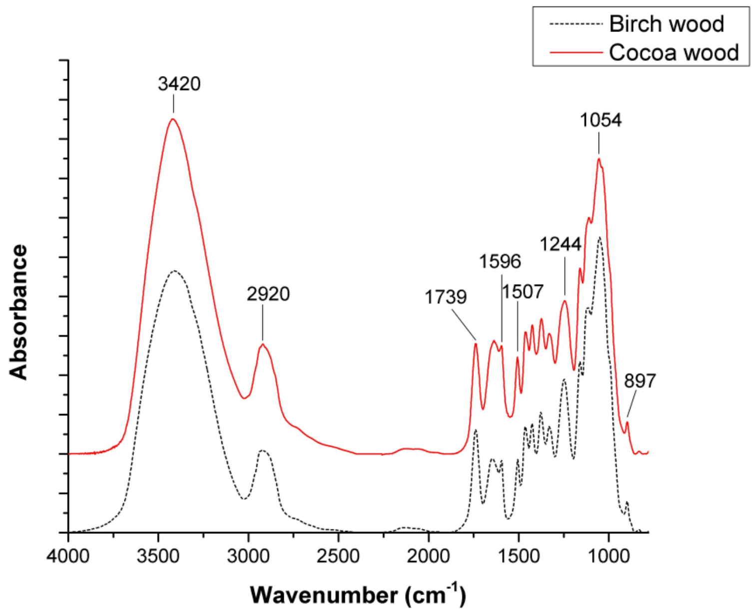

Fig. 3. FTIR absorbance spectrum of cocoa branch wood and birch wood

The FTIR spectra indicated that the cocoa branch wood and birch wood samples contained guaiacyl-syringyl type lignin. However, the ratio of the bands at 1595, 1463, 1424, and 1331 cm^{-1} indicated some differences in both lignin guaiacyl/syringyl ratios. Guaiacyl lignin has a greater tendency to condense, whereas syringyl lignin's linear configuration enables it to dissolve more readily during cooking. Higher absorbance intensity values at 1739 and 1244 cm^{-1} (C-O stretch of acetate group) indicate that birch wood contains more acetyl groups and xylan, the main source of acetyl groups in hardwood, than cocoa branch wood. The chemical analyses in Table 1 confirm these estimations.

Low lignin, mineral, extractive content, and high pulp yield are important chemical characteristics of biomass for the paper industry.

Table 1. Chemical Characterization of Cocoa and Birch Wood

Compound	Amount (%)	
	Cocoa	Birch
Extractives	1.1 ± 0.2	1.6 ± 0.3
Glucan	43.5 ± 0.1	37.8 ± 0.1
Xylan	13.7 ± 0.5	22 ± 0.1
Galactan	0.17 ± 0.04	0.83 ± 0.05
Arabinan	1.1 ± 0.1	0.7 ± 0.1
Mannan	6.0 ± 0.4	1.6 ± 0.5
Ash	0.11 ± 0.01	0.6 ± 0.01
Acetic acid	6.2 ± 0.1	6.7 ± 0.3
Klason lignin	26.2 ± 0.5	18.9 ± 0.4

The chemical composition of cocoa branch wood samples showed higher lignin content, reaching almost 26% compared to the traditionally used in the paper industry birch wood samples (19%; Table 1) and eucalyptus (20-25%), described by other authors (Rencoret *et al.* 2006; Evtuguin and Neto 2007; Vieira *et al.* 2021). The birch branch wood has a lignin content of 21% (Raisanen and Athanassiadis 2013). Cocoa branch wood contains lower concentrations of minerals and extractives than birch wood, with 0.11% minerals and 1% extractives compared to birch's respective levels (Table 1). The content of acetone extractives in cocoa branch wood is much lower than in other hardwood and fruit tree branches (López *et al.* 2000; González *et al.* 2013; Gülsoy *et al.* 2015; Haapala *et al.* 2017). Based on the glucose content, the amount of cellulose in the wood sample could be calculated (Ji *et al.* 2020). Glucose and cellulose contents for cocoa branch wood were found to be 43.5% and 39%, while for birch wood, it is 37.8% and 34%, but pulp yield is 42% and 48%, respectively (Tables 1 and 2). Eucalyptus wood, a commonly used resource for pulping, has a pulp yield between 48% and 54% (Rencoret *et al.* 2006; Evtuguin and Neto 2007; Vieira *et al.* 2021). Cellulose content is the main factor that affects the pulp yield. However, a high content of hemicelluloses, especially xylan, can also increase pulp yield (Evtuguin and Neto 2007). As shown in Table 1, the hemicellulose content in birch wood is higher, increasing the pulp yield. Research conducted by other authors has indicated that eucalyptus xylan exhibits a notably higher degree of polymerization and structural association with other polysaccharides compared to xylans from other types of wood. This characteristic leads to a marked reduction in the solubility of xylan during the kraft cooking process, increasing pulp yield (Evtuguin and Neto 2007). The amount of xylan dissolved during kraft pulping is higher for birch than eucalyptus (Evtuguin and Neto 2007). The relatively high content of xylose and acetyl groups (Table 1) in the cocoa branch wood creates interest in releasing and recovering furfural and acetic acid (Puke *et al.* 2021a,b) before kraft pulping. The lignin content of the pulp is similar to that of both birch and cocoa (Table 1). The obtained cocoa pulp has a high cellulose content and a small content of lignin and hemicelluloses.

Table 2. Characterization of Cocoa Branch Wood Pulp Compared to Birch Pulp

	Pulp Yield (%)	Klason Lignin in Pulp (%)	Brightness ISO (%), unbleached	Brightness ISO (%), bleached
Cocoa	43 ± 2	1.8 ± 0.2	38.8 ± 1.6	60.6 ± 1.7
Birch	49 ± 3	1.8 ± 0.2	34.4 ± 1.8	63.2 ± 1.3

As can be observed from Table 2, the cocoa branch pulp had a high cellulose content, considering that the cellulose content in the cocoa branch was 39%, the pulp yield was 42%, and there was a small content of lignin and hemicelluloses. The pulp yield of cocoa branch wood was comparable to the pulp yield of the other fruit tree branch wood (López *et al.* 2000; González *et al.* 2013; Gülsoy *et al.* 2015). Since lignin is the pulp component that reduces pulp brightness, this is further evidenced by the brightness results in Table 2, which indicates that the unbleached cocoa branches pulp possessed a greater brightness than birch pulp. Figure 4 visually compares pulp handsheets derived from birch and cocoa branches before and after bleaching. In contrast to the brownish-yellow color of the birch wood pulp, which remains unchanged even after bleaching, the cocoa branch pulp exhibited a distinctly gray hue.

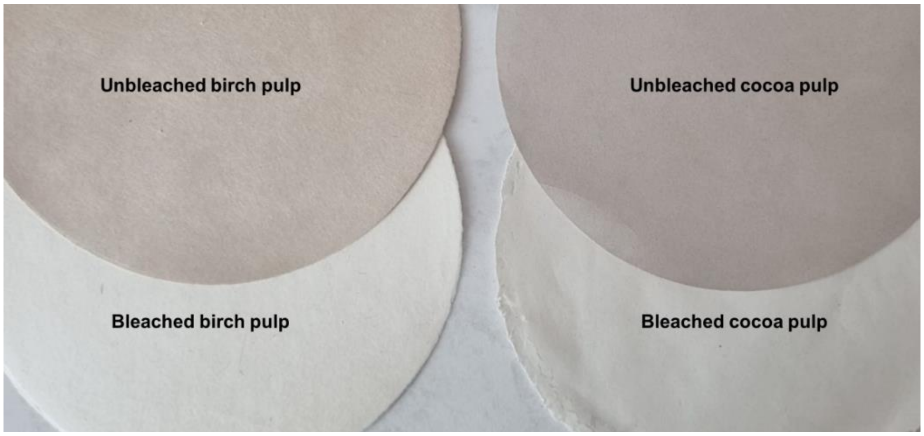

Fig. 4. Visual representations of unbleached and bleached cocoa branch pulp handsheets

Figure 5 presents SEM images of both bleached (a, b) and unbleached (d, e) cacao pulp fibers, as well as bleached (c) and unbleached (f) torn paper samples. The analysis revealed that bleaching had a minimal impact on the length and width of the fibers. There was also no noticeable difference between the bleached and unbleached fibers in the areas where the paper had been torn (Fig. 5c and f). However, the vessels exhibited a noticeable shrinkage following the bleaching process (Fig. 5b, arrow) compared to the unbleached sample (Fig. 5e, arrow).

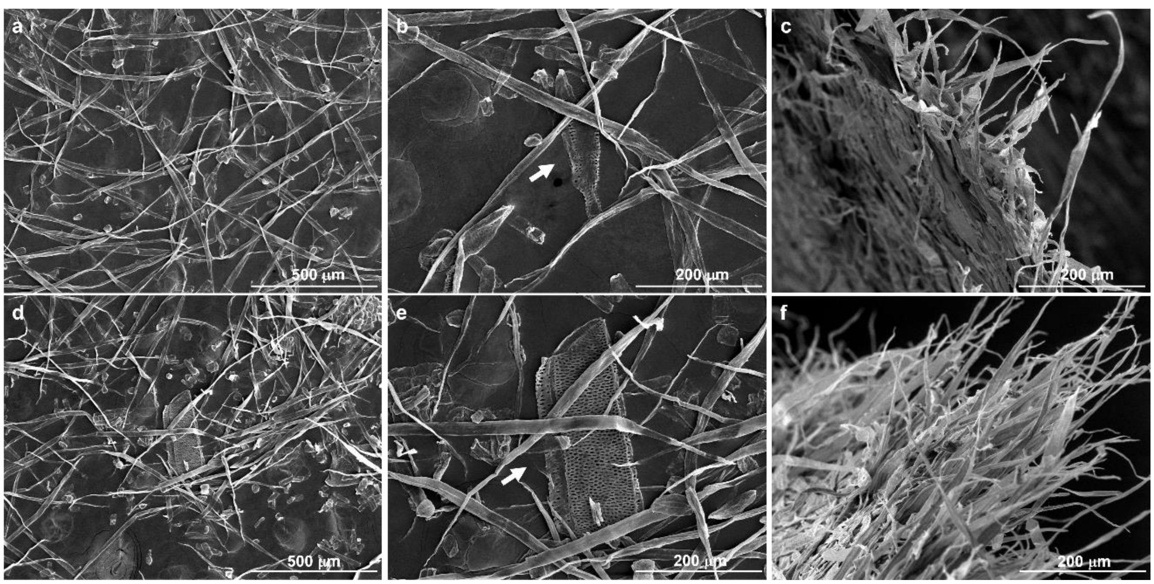

Fig. 5. Scanning electron micrographs of bleached (a, b) and unbleached (d, e) cocoa fibers and bleached (c) and unbleached (f) torn paper samples. Arrows indicate vessels.

Fiber dimensions of cocoa branch pulp showed hardwood-appropriate results (Table 3 and Fig. 6). Fibers of cocoa branch pulp were found to be shorter than birch fibers but comparable to eucalyptus and fruit tree pulp fibers (Table 3) (López *et al.* 2000; Gülsoy *et al.* 2015). The fiber width of cocoa branch pulp was found to be equal to the eucalyptus pulp fiber but smaller than fibers of birch and other fruit tree pulp (López *et al.* 2000; Gülsoy *et al.* 2015).

Table 3. Fiber Dimensions of Cocoa Branch Pulp and Birch Pulp

	Fiber Length (mm)	Fiber Width (µm)	Shape (%)
Unbleached cocoa	0.72 ± 0.01	15.5 ± 0.10	91.3 ± 0.1
Bleached cocoa	0.67 ± 0.02	15.0 ± 0.12	91.5 ± 0.2
Unbleached birch	0.94 ± 0.10	19.7 ± 0.3	91.0 ± 0.2
Bleached birch	0.89 ± 0.09	19.2 ± 0.4	91.6 ± 0.4

As depicted in Fig. 6, the Gaussian distribution of pulp fibers from cocoa branches exhibited a narrower profile than the birch fibers, indicating a more concentrated distribution in length and width. It is well-known that a homogeneous mass of fibers or a narrow distribution of fiber lengths provides a stronger paper, even if the fibers are short (Pulkkinen 2010). Bleaching did not statistically significantly affect the dimensions of birch fibers. However, bleaching significantly reduced fiber length for cocoa branch pulp, although the absolute numbers are similar for bleached and unbleached samples. This can be explained by the narrow distribution of fiber dimensions.

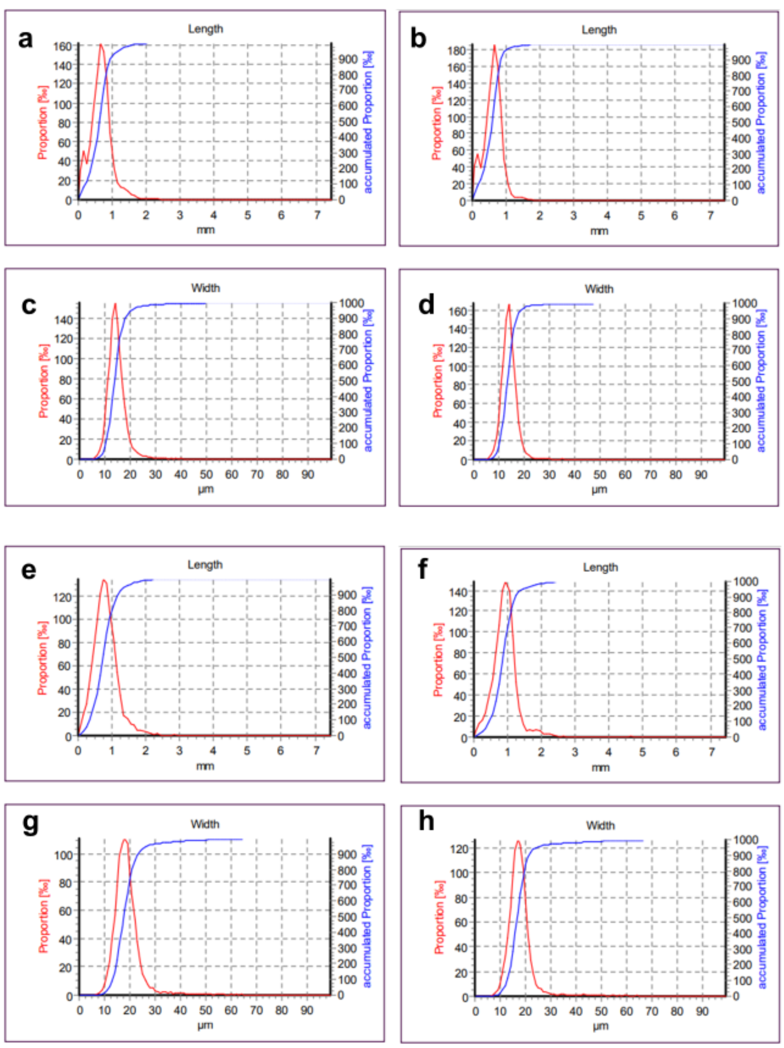

Fig. 6. Fiber length (a; b; e; f) and width (c; d; e; f) distribution of unbleached (a; c) and bleached (b; d) cocoa branch wood and unbleached (e; g) and bleached (f; h) birch wood

Handsheets of unbeaten cocoa branch pulp showed higher mechanical properties (Fig. 7) compared to unbeaten birch and eucalyptus pulp (Shackford 2003; Resquin *et al.* 2006; Pulkkinen 2010) with equal Shopper-Rigler degree (Table 4), reaching a tensile index value equivalent to unbeaten softwood pulp (Sable *et al.* 2017).

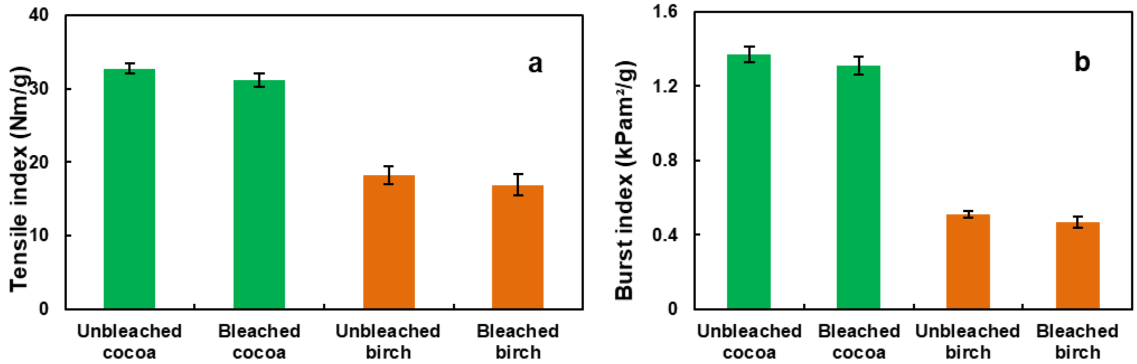

Fig. 7. Mechanical properties of bleached and unbleached cocoa pulp *vs.* pulp of birch: a) Tensile index of the handsheets; b) Burst index of the handsheets

Table 4. Handsheet Paper Density and Bulk of Cocoa Branches and Birch

	Paper Density (g/cm^3)	Bulk (cm^3/g)	°SR
Unbleached cocoa	0.53 ± 0.01	1.84 ± 0.02	16 to 18
Bleached cocoa	0.51 ± 0.01	1.95 ± 0.03	
Unbleached birch	0.38 ± 0.01	2.63 ± 0.03	17 to 19
Bleached birch	0.35 ± 0.02	2.86 ± 0.02	

The tensile and burst indexes of cocoa branch pulp handsheet were 2 and 2.5 times higher than those of birch and eucalyptus traditionally used in the paper industry. Bleaching had a negligible impact on the paper's mechanical properties.

Because of its great mechanical properties, cocoa branch pulp could be used, *e.g.*, for packaging material development. The high mechanical properties of cocoa branch pulp handsheet are justified by the tensile index, which is directly dependent on paper density or inversely proportional to the bulk (Kibblewhite *et al.* 2000). Table 4 shows the corresponding paper density and bulk of cocoa branch pulp compared with birch pulp. It was observed that fiber dimensions and arrangements in handsheets and fiber length distribution influence paper density. The more homogeneous the dimensions of the pulp fibers, the higher the density and the higher the mechanical properties.

CONCLUSIONS

1. Results of the study confirm that despite having a high lignin content, cocoa (*Theobroma cacao* L.) branch wood biomass is suitable for cellulose production using the kraft process.

2. Chemical composition and fiber dimensions of cocoa branch wood show hardwood-appropriate results.
3. Cocoa branch kraft pulp fiber has a narrow fiber length distribution, leading to high apparent density and increased mechanical strength. The tensile and burst indexes of cocoa branch pulp handsheet are 2 and 2.5 times higher than wood pulp traditionally used in the paper industry.
4. Cocoa branch pulp could be used to develop paper packaging materials like paper bags or the outer layer of corrugated paperboard.

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