Characterisation of the lignocellulosic properties of pruning waste from tree species with the potential for furniture production in Acaraú, Ceará[1]

José Dionis Matos Araujo[2], Men de Sá Moreira de Sousa Filho[3], Adriano Lincoln Albuquerque Matos[3], João Alencar de Sousa[3], Francisco Pereira Marques[4], Alisson Moura Santos[5], Antonio Marcos Esmeraldo Bezerra[6]

ABSTRACT - The aim of this study was to characterise the lignocellulosic fractions of pruning waste from tree species showing potential for furniture production in Acaraú, Ceará. The experiment was conducted in a completely randomised, split-plot design, with the plots corresponding to two water regimes (irrigated for one year and irrigated for three years) and the sub-plots to eight tree species, with three replications. The chemical attributes were determined after seven years. Except for the insoluble lignin content, there was a significant effect from the different species in terms of the chemical properties of the wood. The results for the lignocellulosic fractions show the possibility of developing technological ways of adding value to these waste products, especially considering their lignin content. The different species also showed potential for use in producing cellulose pulp, with the exception of *Handroanthus impetiginosus* due to its high ash content. The results of this study offer new perspectives for future research into the use of forestry waste.

Key words: Chemical characterisation. Native. Exotic.

INTRODUCTION

The state of Ceará is regionally important for the production of wooden furniture, with more than 300 companies directly generating over 3,000 jobs (De Arruda Coelho, 2022). However, raw materials in the form of wood mainly come from other parts of the country, resulting in additional logistical costs for the furniture industry as well higher production costs.

As one alternative for dealing with this situation, the introduction of different tree species, both exotic and native, with the potential for providing wood for the local furniture industry, has been studied in the irrigated perimeter of Baixo Acaraú in Ceará. Reforestation using native species can potentially restore areas that were once occupied by forests and reduce the pressure for deforestation in areas of native forest. (Sist, 2021).

One result of this strategy is the waste generated from lignocellulosic fractions by such processes as pruning and tree-felling that could be used by the furniture industry as raw material.

Wood is characterised as a material consisting essentially of biopolymers (cellulose, hemicellulose, and lignin) organised in a complex three-dimensional structure whose physical and mechanical properties are mainly determined by the cellulose, hemicellulose and lignin that make up the cell wall and maintain its integrity (Shi *et al*, 2022).

Lignin is the second largest component of plant cell walls, making it the second most important renewable natural resource after cellulose (Chen *et al*, 2020). Its significance is further enhanced by the fact that it is the most important biopolymer with aromatic rings and active hydroxyls in its structure, and is largely composed of three basic units: guaiacyl, syringyl, and p-hydroxyphenyl (Yuan *et al*, 2023).

Like other biorenewable polymers, lignin offers several advantages, including its antioxidant and antimicrobial properties, its abundance as a by-product of industrial waste, and its biodegradability. In addition, lignin improves resistance in the cell walls of cellulosic materials, protecting the cell wall from biochemical stress and inhibiting the enzymatic degradation of other components. Compared to cellulose, hemicellulose, and other polysaccharides, lignin is reported to be resistant to most types of biological attack (Doherty *et al*., 2011). In this respect, Mattos (2017) notes in particular that lignocellulosic biomass, available in the form of agricultural and agro-industrial waste, stands out as an interesting alternative for the development of new materials.

The most common natural biopolymer in the environment is cellulose, with an estimated annual production of one trillion tonnes. Cellulose is a polymer made up of glucose molecules connected by β-1,4 glycosidic bonds (Yan *et al*, 2023). Because it is renewable, non-toxic, biocompatible, chemically and thermally stable, of low-cost, and has versatile physical and mechanical properties, cellulose is the basis for studies aimed at developing substitutes for petroleum-based polymers, the production of thickeners and emulsion stabilisers for the food industry, and the production of plastic or paper packaging and moulded pulp for the chemical industry, for medicine, cosmetics, construction, etc (Aziz *et al*, 2022).

Hemicellulose is a heterogeneous group of polysaccharides with degrees of polymerisation well below those of cellulose (80 to 200), and a chemical composition that varies depending on the plant species, type of cell, location in the plant, and stage of development. The most common sugars in the composition of hemicellulose are pyranose, furanose, d-xylose, d-mannose, d-glucose, d-galactosyl, and l-arabinose, as well as galacturonic acid and glucuronic acid. Hemicellulose is used in the production of various materials for the chemical, food and medical industries, such as films, hydrogels, and emulsifiers (Rao *et al*, 2023).

Lignocellulosic fibres have recently emerged as an economic and ecological alternative for use as reinforcements and fillers in composites with virgin or recycled polymers (Mattos, 2017). Several authors have discussed the potential application of lignin, such as its use in panels with antifungal properties (Zhang *et al*. 2015). In their study, Peng and Chen (2011) also synthesised lignin-based hydrogels using lignin with polyurethane ionomers to study the release of ammonium sulphate. They found that these hydrogels could potentially be used as coating materials to produce a controlled-release fertiliser with various agricultural applications.

The aim of this study was to chemically characterise the lignocellulosic fractions of pruning waste from species being tested in the furniture industry, with a view to evaluating possible ways of adding value, particularly for the lignin.

MATERIAL AND METHODS

The study evaluated the wood from branches of the following tree species (SIGEN: AF46C91): *Acacia mangium* Willd, *Anadenanthera colubrina* var. *cebil* (Griseb.) Altschu, *Casuarina equisetifolia* L. ex J. R. Forst. & G. Forst, *Handroanthus impetiginosus* (Mart. ex DC.) Mattos), and *Colubrina glandulosa* subsp. *reitzii* (M.C.Johnst.) Borhidi); and from seven-year-old eucalyptus clones: GG 680 (*Eucalyptus urophila* x *Eucalyptus grandis*), VE 38 (*Eucalyptus urophila* x *Eucalyptus camaldulensis*), and VE 41 (*Eucalyptus urophila* x *Eucalyptus grandis*). The wood

was collected from an experimental area run by Embrapa Agroindústria Tropical, in an area of the Baixo Acaraú Irrigated Perimeter bordering the district of Marco, Ceará, located at 3°06'02" S and 40°04'05" W, at an altitude of 56 metres. According to the Köppen classification, the climate in the region is type Aw' (tropical rainy). There is a marked change between the rainy season (January to May) and the dry season (June to December). The average annual rainfall varies around 900 mm, with an average annual temperature of 28.1 °C, average annual relative humidity of 70%, average annual evaporation of 1600 mm, sunshine of 2,650 h/yr, and average wind speed of 3.0 m/s (DNOCS, 2016). The soil in the experimental area was classified as a quartzarenic Neosol (EMBRAPA, 2013), with the following characteristics in the 0 to 50 cm layer: textural class, sand; soil moisture at 0.03 MPa, 4.38%; moisture at 1.5 MPa, 2.95%; pH, 6.1; EC, 0.19 dS.m^{-1}; organic matter, 9.0 g. Kg^{-1}; P, 19.9 mg dm^{-3}; and 14.65, 6.45, 1.05, 3.35 and 5.4 mmol$_c$.dm^{-3} of Ca^{2+}, Mg^{2+}, K$^+$, Na$^+$, and H$^+$+Al3$^+$, respectively.

The experiment was set up in an area that was divided into two sub-areas (plots), one in which irrigation was suspended after the first 12 months (one-year irrigation regime) and the other in which irrigation was suspended after three years (three-year irrigation regime). During the first 12 months the entire area was irrigated daily by micro-sprinkler with an irrigation depth of 2.7 mm.day^{-1}. Following this period, the irrigation frequency in the three-year regime was changed to two days and the irrigation depth to 5 mm.day^{-1}.

The species were planted in experimental sub-plots measuring 6 x 28 m, comprising three rows of 15 plants/row, with the first and third rows considered borders together with the first and last plants in the central row. A spacing of three metres was used between rows and two metres between plants.

Three trees were selected per sub-plot, each with a good phenotypic profile, and branches of different diameters were collected from each plant to form a composite sample for chemical characterisation of the wood.

The branches were ground in a knife mill under laboratory conditions and passed through a 20-mesh sieve. The chemical attributes of the wood were extractives (TAPPI. T204 cm-97, 1997), ash (TAPPI. T211 om-02, 2002), insoluble lignin (TAPPI. T 222 om-22, 2002), alpha-cellulose (TAPPI. T203 cm-99, 2009), and hemicellulose, obtained from the difference between the holocellulose and alpha-cellulose content. The holocellulose content was determined using the method described by Yokoyama *et al.* (2002).

The lignin in the samples of *Acacia mangium*, *Anadenanthera colubrina*, *Casuarina equisetifolia*, *Colubrina glandulosa*, GG 680, VE 41, and VE 38 was extracted using the acetosolv method, as described

by Leitão *et al* (2019). The samples were pre-treated in a steam explosion reactor at 168 °C for 10 min. The acetossolv extraction took place in a high-pressure reactor at 190 °C for 26 min, using 1 L of a 75% aqueous solution of commercial ethyl alcohol with 0.5% sulphuric acid per 100 g of sample. This stage results in a black liquor, which is centrifuged at 700 rpm to extract the remaining solids, and the liquid phase filtered twice in a filter press of 8-μm filter paper to obtain the acetosolv lignin.

The Fourier transform infrared (FTIR) spectra of the lignin samples were obtained using a Spectrum Two FT-IR spectrophotometer (PerkinElmer) employing KBr pellets with a sample concentration of 5% (w/w). The spectra were obtained at wavelengths between 4000 and 400 cm^{-1} at a resolution of 1 cm^{-1} using the arithmetic mean of 32 readings. The FTIR data was processed and analysed using the Origin v9.4 software.

A thermogravimetric analysis of the lignin samples was carried out using an STA 6000 thermal analyser (PerkinElmer). All the measurements were taken in a nitrogen atmosphere at a gas flow rate of 50 mL.min^{-1}, heating from 30 °C to 900 °C at a heating rate of 10 °C.min^{-1}. Sample weights of approximately 10 mg were used.

The experiment was conducted in a completely randomised, split-plot design, with the plots corresponding to the two water regimes (one year of irrigation and three years of irrigation) and the subplots corresponding to the eight species. Three plants were selected and distributed over three replications.

The data were submitted to analysis of variance to check for isolated effects and interaction between the factors, and the mean values were compared by Scott-Knott test (p < 0.05). The statistical analyses were carried out using the Analysis of Variance for Balanced Data (SISVAR) software, developed by Ferreira (2008).

RESULTS AND DISCUSSION

Statistical analysis of the water regimes and species

A completely randomised design was used to check for variations in the chemical characteristics of the wood using two water regimes and eight different species. For this purpose, the ash content, total extractives, insoluble lignin, hemicellulose and alpha-cellulose of the samples were determined.

Changing the irrigation regime from 12 to 36 months did not significantly affect the chemical composition of the eight materials under evaluation (Table 1). This suggests that an irrigation strategy of up to 12 months is effective in maintaining the chemical quality of the wood while saving

water. Similar results were found by Barbosa *et al.* (2014) studying a six-year hybrid of *E. urophila* x *E. grandis.*, and by Moulin *et al.* (2015), who, after 6 and 12 months, assessed the composition of two eucalyptus clones, with and without irrigation, and found significant differences in the extractive content.

Species had a significant effect on the chemical properties of the wood, except for the insoluble lignin content (Table 1). The ratio between the main chemical constituents of the wood varies between genera, between species, and within any one tree, and is also affected by variations in the microclimate, soil, rainfall, fertiliser and age of the tree, among others (GOMIDE and COLODETTE, 2007). Differences in chemical composition between different clones and tree species have also been observed by other authors (MOULIN, 2015; HOUSSAIN, 2023).

Chemical characterisation of the wood

Each of the species under study had a high ash content regardless of the water regime, with the exception of *Casuarina equisetifolia* (0.93%). *Handroanthus impetiginosus* had the highest ash content (2.28%) of all the species under study (Table 2). Medeiros Neto *et al.* (2012) found an ash content of 0.87% in *Handroanthus impetiginosus*, well above the average found in the present study. Villaseñor Araiza and Rutiaga Quinõnes (2000) found an ash content of 0.5% for sapwood and 1.5% for heartwood in *Casuarina equisetifolia*, similar to the values found in the present study. In the energy sector, ash values below 1% are considered ideal (SANTOS *et al.*, 2011), and only *Casuarina equisetifolia* showed no values above this limit. High levels of minerals in the wood result in a high percentage of ash in the charcoal, which is detrimental when the charcoal is used in steelmaking (ANDRADE, 1993).

In the cellulose sector, the range accepted by industry varies from 0.2% - 1.1% (FOELKEL, 2011), with only *Casuarina equisetifolia* and the VE - 38 clone meeting this criterion. Chaves *et al.* (2013) state that the ash content is inversely related to the calorific value, since, when burning biomass, volatile materials evaporate quickly, reducing the residence time of the fuel in the combustion unit, and potentially contributing to low energy efficiency.

Handroanthus impetiginosus and *Acacia mangium* had the highest extractive content, 2.56% and 2.32% respectively, differing statistically from the other species (Table 2). However, for energy purposes, the total extractive content is below that considered suitable for the sector, around 4.5% to 6.5% (TRUGILHO *et al.*, 2001). The total extractives have a high calorific value and are positively correlated with the gravimetric charcoal yield (SANTOS *et al.*, 2011). The low values for extractives, especially in the eucalyptus clones, is of great importance in cellulose production since a high level of total extractives is detrimental to the process. During the production process, the extractives can agglomerate, and form pitch deposits, which reduces the useful life of the equipment, increases alkali consumption, and lowers the quality of the final product by reducing the absorbency of the pulp (SILVESTRE, 1999; D'ALMEIDA *et al.*, 2013; COLODETTE; GOMES, 2015). For the eucalyptus clones, the extractive content is within the range reported by other authors, between 1.09% and 4.28% (FERREIRA *et al.*, 2006; GOMIDE *et al.*, 2005; TOLFO *et al.*, 2005; SÃO TEAGO, 2012). For *Handroanthus impetiginosus*, the extractive content is lower than that found by Medeiros Neto *et al.* (2014), who recorded a content of 6.88%. Paschoal Neto *et al.* (2005), studying 7-year-old *Acacia mangium*, obtained an extractive content of 4.06%, which is higher than

Table 1 - Summary of the analysis of variance and coefficient of variation (CV) for pruning ash (PASH), total extractives (TOT EXT), insoluble lignin (INS LIG), hemicellulose (HEMI), and alpha-cellulose (ALPHA) in five tree species and three eucalyptus clones grown under two water regimes (irrigated for 1 year and irrigated for 3 years) seven years after planting, Acaraú, Ceará, 2019

SV	DF	MS				
		PASH	TOT EXT	INS LIG	HEMI	ALPHA
Regime (A)	1	0.0124^{ns}	0.0000^{ns}	1.0086^{ns}	2.6583^{ns}	0.0676^{ns}
Residual (a)	4	0.0548	0.4598	4.1836	3.8616	0.7881
Species (B)	7	1.1564^{**}	1.7891^{**}	$11.1215ns$	29.9318^{**}	16.9364^{**}
AxB Interaction	7	0.0913^{ns}	0.2062^{ns}	5.7007^{ns}	11.2774^{ns}	3.6915^{ns}
Residual (b)	28	0.0891	0.1724	4.7810	5.7675	5.0069
CV(a) (%)	-	16.13	39.56	7.85	6.23	2.44
CV(b) (%)	-	20.58	24.22	8.39	7.62	6.14

** Significant at 1% probability by F-test; ns Not significant by F-test

Table 2 - Mean values for pruning ash (PASH), total extractives (TOT EXT), insoluble lignin (INS LIG), hemicellulose (HEMI) and alpha-cellulose (ALPHA) in five tree species and three eucalyptus clones seven years after planting, Acaraú - Ceará, 2019

Species/Clone	PASH	TOT EXT	INS LIG	HOLO	
				ALPHA	HEMI
	------------------------------- % -------------------------------				
Acacia mangium	1.07 c	2.32 a	27.47 a	37.81 a	28.94 b
Anadenanthera	1.70 b	1.88 b	23.79 a	37.96 a	29.47 b
Casuarina	0.93 c	1.95 b	26.04 a	37.98 a	32.34 a
Handroanthus	2.28 a	2.56 a	27.73 a	33.07 b	28.69 b
Colubrina	1.63 b	1.39 c	26.43 a	35.15 b	31.52 a
GG 680	1.59 b	1.38 c	25.20 a	36.20 a	34.10 a
VE 38	1.11 c	1.11 c	26.79 a	36.44 a	33.96 a
VE 41	1.30 c	1.13 c	24.79 a	36.74 a	33.18 a

Mean values followed by the same letter in a column do not differ by Scott-Knott test at a level of 5%

the value found in the present study. However, the discrepancy between these results may be due to differences in the extraction methods or the types of solvents used.

The lignin content found in *Casuarina equisetifolia* (26.04%) is in line with the levels proposed by Vidal and Da Hora (2011) for conifers, ranging from 19% to 33%, and is also in line with the lignin content found for the other species under study (23.79% to 27.73%), where the authors propose levels in hardwoods that range from 13% to 31%.

In the energy sector, the values for insoluble lignin found in the present study are considered intermediate (23.79% to 27.73%) according to Trugilho *et al.* (2001). The sector requires high values for this compound, as it has a positive correlation with higher calorific values, gravimetric charcoal yield, and the fixed carbon content (BRITO and BARRICHELO, 1977; VITAL *et al.*, 1994; SANTOS *et al.*, 2011).

In addition, the lignin content found in the present study is acceptable to the pulp and paper sector, as falls within the 27.1% to 31.3% range (JARDIM *et al.*, 2017). However, as mentioned above, the high ash content of *Handroanthus impetiginosus* (2.28%) makes it impossible to use (Table 2); it does however have the potential for use in the production of other nobler forms of cellulose with higher added value, such as microfibrils and nanocrystals, given that the ash content did not hamper production of these materials from palm oil waste (Marques *et al.*, 2020; Souza *et al.*, 2016). Furthermore, it is worth noting that the lowest possible lignin content is best for cellulose production as it facilitates wood pulping (GOMES *et al.*, 2008).

Characterisation of the lignin

Fourier transform infrared spectroscopy (FTIR) was used to determine qualitatively the monomeric composition of the lignin. The polymeric structure of lignin is made up of three monomers whose basic difference lies in the absence or presence of methoxy group(s) ($-OCH_3$) linked in the ortho position of the aromatic lignin rings. A p-hydroxyphenyl (H) unit is one which has no methoxy group attached to the aromatic ring. A guaiacyl unit (G) has only one methoxy group, while a syringyl unit (S) has two methoxy groups. The presence of these groups on the aromatic ring can hinder the application of lignin by occupying reactive sites on the ring. However, it depends on whether the application requires reactions in the aromatic rings, such as the application of phenolic resins of the lignin-phenol-formaldehyde type (PINHEIRO *et al.*, 2017).

The FTIR spectra (Figure 1) showed that the wood lignins in the present study were made up of G (guaiacyl) and S (syringyl) units (Faix, 1991). The shoulder at 1268 cm^{-1} corresponds to the vibrations of the aromatic rings of the G units from stretching of the C=O bonds. Furthermore, the signal at 1218 cm^{-1} is typical of lignin made up of G units. The band at 855 cm^{-1} confirms the presence of G units via the out-of-plane vibrations of the C-H bonds. In addition, the signals at 1325 and 1116 cm^{-1} indicate the presence of S units.

In addition, thermogravimetric analysis and differential thermogravimetry (TGA/dTG) were used to determine the thermal stability of the lignins. Thermal stability is an important parameter for lignin, as some applications require the use of temperature. The TGA

spectra (Figure 2) showed two thermal events typical of lignins. The first event under 100 °C is related to the possible presence of moisture or low molecular weight structures.

The second event is related to the thermal degradation of the lignin structure. The samples showed good thermal stability (Table 3) starting at temperatures greater than 200 °C (T$_{onset}$) with the maximum degradation temperature (T$_{max}$) occurring around 360 °C, which is a typical lignin event.

Char yield (CY) is the amount of material remaining at the end of combustion, and is related to the ability of the material to retard or extinguish flames during combustion. The use of lignins with this characteristic when preparing new materials may afford the materials flame retardant properties. Materials with a CY greater than 26% are considered self-extinguishing (KREVELEN, 1975); as shown in Table 3, the lignins in the present study have a CY of more than 26%. The abovementioned characteristics of the lignins make them possible candidates for technological application, however a more in-depth study of other characteristics, such as molecular weight, is required.

The following uses have been applied to lignin: the production of phenolic resin, in animal feed, use as dispersants, in biocomposites, as additives, and in the

Figure 1 - FTIR spectra of acetosolv lignins from VE 38, GG 680, ACA (*Acacia mangium*), AND (*Anadenanthera colubrina*), and COL (*Colubrina glandulosa*)
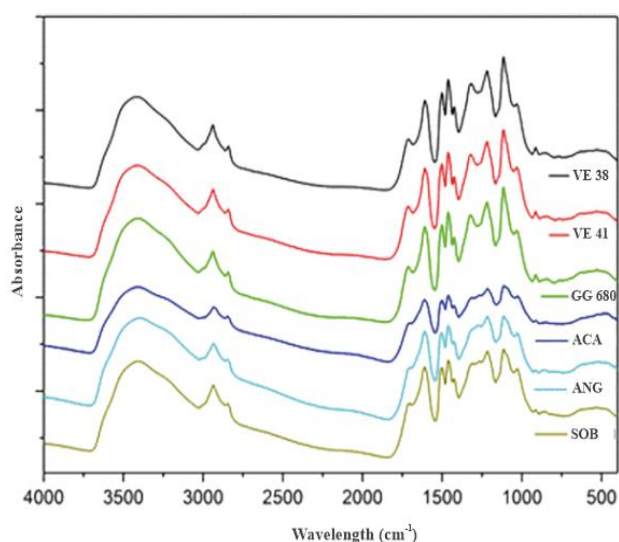

Figure 2 - TGA analysis of the acetosolv lignins from VE 38, VE 41, GG 680, ACA (acacia mangium), AND (*Anadenanthera colubrina*), CAS (*Casuarina equisetifolia*), HAN (*Handroanthus impetiginosus*), and COL (*Colubrina glandulosa*)
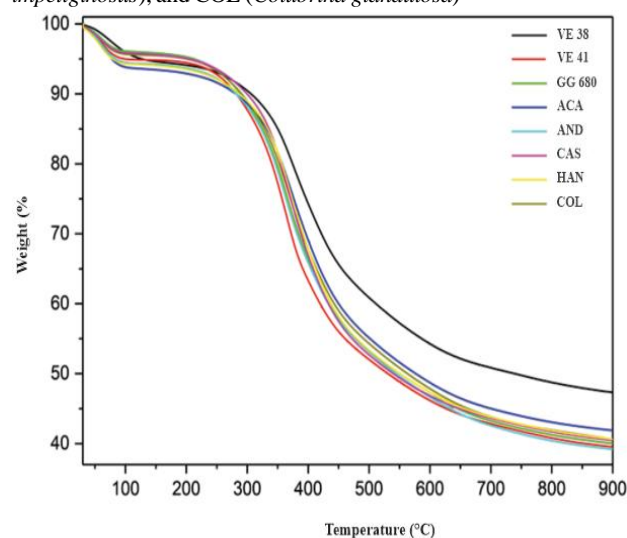

Table 3 - Initial degradation temperature (T$_{onset}$), maximum degradation temperature (T$_{max}$), and char yield at 900°C for the lignins under study

Sample	Tonset (°C)	Tmax (°C)	CY (%)
VE 38	228.6 ± 2.5	372.3 ± 5.4	44.1 ± 4.6
VE 41	215.0 ± 4.7	366.3 ± 2.4	41.3 ± 2.5
GG 680	213.8 ± 6.4	363.7 ± 2.2	39.3 ± 1.1
ACA	215.6 ± 1.6	370.8 ± 4.1	40.7 ± 2.0
AND	220.1 ± 3.3	373.9 ± 8.6	41.7 ± 2.2
CAS	202.1 ± 4.6	368.1 ± 1.6	40.4 ± 1.4
HAN	208.4 ± 1.0	373.1 ± 0.9	41.6 ± 1.1
COL	221.4 ± 5.3	371.4 ± 5.5	41.6 ± 1.3

ACA (*Acacia mangium*); AND (*Anadenanthera colubrina*); CAS (*Casuarina equisetifolia*); HAN (*Handroanthus impetiginosus*); COL (*Colubrina glandulosa*)

formation of polymer blends, surfactants, thickeners, fine chemicals, ceramic products, pesticides, as additives in concrete and cement, and many others (VISHTAL; KRASLAWSKI, 2011).

Lignin can be used in the chemical industry in more noble applications, such as in cosmetic formulations (VINARDELL *et al.*, 2008), as a substitute for phenol in the production of phenol-formaldehyde resins, as a precursor for carbon-based materials (activated carbon) (DUVAL; LAWOKO, 2014), as adsorbents (DUVAL; LAWOKO, 2014), as a copolyol in the synthesis of polyurethanes (BERNARDINI *et al.*, 2015), etc.

The greater hemicellulose content (32.34% to 34.10%) and good percentage of alpha-cellulose (36.20% to 37.98%) that result in a high level of holocellulose (68.54% to 72.08%) in *Casuarina equisetifolia* and the eucalyptus clones, make these species suitable for the production of cellulose pulp. The holocellulose content is in the same range as found in older wood in *Eucalyptus urophylla* x *Eucalyptus grandis* hybrids. For example, Ferreira *et al.* (2006) found values of 64.4% in 7-year-old trees, Alencar *et al.* (2001) obtained a content of 67.54% after six years, and Gomide *et al.* (2005), from 64.5% to 70.2% after seven years.

CONCLUSIONS

1. The results show that the strategy of using irrigation during the initial 12 months only was sufficient for maintaining the quality of the wood in terms of its chemical composition;

2. On the other hand, the analysis of the chemical composition and subsequent extraction of lignin acetossolv from the resulting materials allows a positive assessment of the potential for utilising the waste from cultivated forest species in the state of Ceará to produce both chipboard panelling from the lignocellulosic fibres and resins based on the lignin produced by these materials.

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