Research Article
doi: 10.1590/01047760202330013427
vol(30), 2024

CERNE

Production and characterization of wood biochar from energetic forests by pyrolysis in a horizontal screw reactor

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TECHNOLOGY OF FOREST PRODUCTS

ABSTRACT

Background: *Acacia mearnsii* and *Eucalyptus dunnii* are species planted mainly for tannin and cellulose production. However, its woods have characteristics favorable to their use for energy generation, being commercialized in the form of firewood, chips or charcoal too. Biochar is a stable form of carbon that can be obtained by converting small particles of wood in the pyrolysis process and that can be used as solid fuel, among other applications as a soil conditioner. In this study, the properties of biochar produced in a horizontal reactor using wood of these species and under milder temperatures were evaluated.

Results: Temperature of 350 °C and the reaction time of 30 minutes promoted an increase in higher calorific value of 33% for *A. mearnsii* and 51% for *E. dunnii*, with gravimetric yield of 39% and 32%, respectively. it was found that the 250 and 300 °C was not efficient to carbonize the wood of both species. An increase in specific surface area is observed with increasing carbonization temperature. Bigger increases were produced at temperatures of 350 °C for *E. dunnii* and 420 °C for *A. mearnsii*, with increase in porosity. At these temperatures, there was an increase in surface area and a decrease in pore size.

Conclusion: In temperatures of 350 °C, the woods of both species provided a good quality biochar with porosity, surface area, pore size and pyrolysis yield suitable for energy use. For soil conditioning use, considering these aspects, suggested to test the appropriate temperatures for each species, between 350 and 420 °C.

Keywords: Black wattle; Dunn's with gum; Forest biomass; Carbonization.

HIGHLIGHTS

Wood biochar is a promising way to value the energetic forest biomass;
Fast-growing species that produce high-density wood can be used for biochar production;
Wood pyrolysis in horizontal reactor using milder temperatures produces good biochar;
Acacia mearnsii and *Eucalyptus dunnii* were suitable for wood conversion into biochar.

HILLIG, E.; GODINHO, M.; PERONDI, D.; MANERA, C.; NEUWALD, O. A. Production and characterization of wood biochar from energetic forests by pyrolysis in a horizontal screw reactor. CERNE, v. 30, 2024, doi: 10.1590/01047760202330013427

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Received: May 17/2024 Accepted: August 14/2024

CERNE
ISSN 0104-7760
UFLA UNIVERSIDADE FEDERAL DE LAVRAS

INTRODUCTION

Acacia mearnsii is fast-growing forest specie planted in a short rotation regime, the first is an important plantation species for tannin production and woodchip exports in South Africa, Australia and Brazil, for bioenergy and pulp and paper production (Griffin et al., 2011). It was found that this species was promising for establishing mixed-species planting with *Eucalyptus urophylla* x *Eucalyptus grandis* hybrid, at the age of 9 years in an experimental area in South of Brazil, presenting gains in biomass production per hectare (Ludvichak et. al., 2022).

Although, initially planted to obtain tannin from the bark, wood started to provide 85% of the income obtained from the plantations (Chan et al., 2015). Other products can be obtained from its wood, such as source of short fibers to produce kraft pulp with this wood has an average basic density of 0.544 g/cm^3 and an average ash content of 0.35% (Giesbrecht et al., 2022).

The *E. dunnii* is planted mainly in the South of Brazil and Uruguay for the cellulose, wood panels and charcoal production, with large increases in consumption of this species for energy (Dobner Jr. et al., 2017; Bentancor et al., 2019). This species was considered suitable for forest implantation in Australia, China and Central and South America (Jovanovic et al., 2000). Ferreira et al. (2018) demonstrated that small adjustments to the nitrogen fertilization regime can increase the productivity of *E. dunnii* plantations in southern Brazil.

Basic density of 0.497 g/cm^3 and calorific value of 19,620 J/g were found for *E. dunnii* wood obtained from six-year-old commercial stands planted in Uruguay (Resquin et al., 2020). On the other hand, the highest biomass per hectare was achieved for the highest planting density for *E. dunnii* (157-193 Mg ha^{-1} for 6660 trees ha^{-1}) and wood density increased with the age of the crop was dependent on the planting location, occurred at Paysandú and not at Tacuarembó (Resquin et al., 2019).

There are several ways to use forest biomass as a source of energy, including its use in natura or its densification (through briquetting and pelleting) in the combustion process; and torrefaction or pyrolysis process for further combustion. The torrefaction occurs at 200 to 300 ºC, also called by some authors mild pyrolysis, and pyrolysis occurs at 300 to 700ºC. These are thermochemical process where biomass is thermally degraded in an inert atmosphere and that involve various reactions that can be endothermic and exothermic (Basu, 2018; Ciuta et al., 2018; Huang et al., 2020).

The main products of the pyrolysis process of a biomass are a fuel gas, a liquid fraction (bio-oil) and a solid fraction (biochar). The pyrolysis process is flexible, and the yields of the products can be controlled according to the operating parameters of the process (heating rate/temperature/residence time of vapors and solids in the reactor) (Tripathi et al., 2016).

In the wood pyrolysis process, carbonization time and temperature are important, but the combination of these two factors, expressed in a heating rate, can determine the characteristics of the charcoal produced (Tancredi et al., 2011). It was verified for *E. grandis* wood that this influence was greater at temperatures between 300 and 350 ºC than between temperatures between 450 and 650 ºC. (Mermoud et al., 2006).

Variables that influence on the yield of charcoal are biomass heating rate, purging gas flow and particle size. Increasing the wood heating rate results in decreased charcoal yield, therefore, low temperature reactions of charcoal formation are favored by low heating rates and the initial charcoal acts as a catalyst for primary biomass decomposition (Somerville and Deev, 2020).

The temperature is the variable of greater influence on the charcoal properties (Dias Jr. et al., 2020). Pyrolysis of different wood specie were done, but, in general the temperatures are higher that 350 ºC. In these temperatures, results show that the nature of the wood has a much greater impact on charcoal properties than the pyrolysis operating conditions (Dufourny et al., 2019).

In general, it was found that the pyrolysis of wood occurs at temperatures above 300 ºC. However, lower temperatures can be used in order to improve its energy properties, in which case it is considered as "torrefaction" or "pre -carbonization" of wood. Pereira et al. (2016) torrefied for 10 min. *Eucalyptus* sp. wood at temperatures ranging from 150 to 300 ºC, concluding that 250 ºC provided wood with higher energy density.

Similar results were found by Rodrigues and Rousset (2015) who used temperatures of 220, 250 and 280 ºC, however, with higher percentage gains in calorific value at higher temperatures due to the longer torrefaction time (60 min). In the pyrolysis of beech wood chips a greater influence of the particle size (0.21–0.50mm, 0.85–1.70mm and 2.06–3.15mm) was observed on the biochar yield at temperatures below 400 ºC (Yu et al. 2018).

For the pyrolysis carried out in these laboratory studies, was used different equipment and the wood was subjected to the process under different conditions of dimensions and format. Reactor type and capacity, pyrolysis temperature, solid residence time, carrier gas flow rate, vapor residence time and the type and size of biomass feedstock were identified as the parameters that most influenced the yields of the biochar produced and its properties (Brassard et al. 2017). The authors described several types of screw reactors used in research around the world, including the one used in this study, classifying them as: a) smaller capacity single screw (less than 1 kg/h); b) higher capacity single screw (1 to 15 kg/h); c) industrial single screw (greater than 15 kg/h); and d) double screw.

Thus, the hypothesis was formulated that *A. mearnsii* and *E. dunnii* are promising species of short-cycle forests that can provide wood suitable for pyrolysis at mild temperatures and biochar with good characteristics for energy use. The objective of this study was to produce and evaluate the biochar resulting from the slow and mild temperatures pyrolysis of these woods through their chemical, energetic and morphological properties.

MATERIAL AND METHODS

Raw material

Woods of *Acacia mearnsii* (Black wattle) and *Eucalyptus dunnii* (Dunn's white gum) were used. A visit was made to companies that use these species for chip production, and the chips were obtained at equilibrium moisture content.

Eucalyptus dunnii wood were obtained from forest in Santa Catarina state. The climate in the region is classified as Cfb according to the Köppen classification. Frosts occur in the winter periods, with records of 27 frosts per year. The average annual temperature is 17.8 ºC and the annual rainfall is 1,841 mm (KLABIN, 2024). Forest was managed in a coppice system and harvested in an average time of 7 years.

Acacia mearnsii wood was obtained from Rio Grande do Sul state where there are approximately 90,000 ha of forest type. According to the Köppen classification, the climate in the region is humid to sub-humid of type Cfa, with rainfall of 1,200 mm to 1,700 mm. The annual average temperature is below 20 °C, reaching 14°C at the highest altitudes (SETA, 2022). The cut cycle used has an average of 7 years and the harvest is carried out with a Harvester in the system of short logs with 2.2 m.

The chips were dried in an oven at 103 °C and then submitted to a knife mill with 3 mm opening sieve, both species in the same grinding conditions. The particles resulting from the milling were used for pyrolysis (Figure 1 and Table 1).

In preparing the raw material, in addition to drying and grinding the chips to obtain particles for pyrolysis, the biochar produced was also milled for scanning electron microscopy, proximate chemical and thermogravimetric analysis (Figure 2).

Biochar production

A horizontal pyrolysis reactor was used (Figure 3). The particle pyrolysis occurred through a 2 meters conveyor screw, with a diameter and pitch both of 195 mm. The material travels 1.34 meters to the biochar discharge outlet in a 40-liter collector. The motor that drives the screw conveyor is controlled by a frequency inverter that allows controlling the feed speed and, therefore, the residence time of the solids in the pyrolysis process.

Table 1: Wood physical characterization.

Material	Chips		Particles	
Species	Bd (g/cm³)	MC (%)	Bk (g/cm³)	Pd (mm)
A. mearnsii	0.550 (0.024)	4.53 (0.51)	0.248 (0.007)	0.270
E. dunnii	0.526 (0.017)	3.38 (0.34)	0.219 (0.002)	0.259

Bd: Basic density; MC: Moisture content; Bk: Bulk density; Pd: Average particle diameter (based on average sample volume).

The reactor body consists of a cylindrical annular combustion chamber with an internal diameter of 200 mm and an external diameter of 300 mm. This chamber is heated by a combustor fed with LPG gas and has 3 zones where temperatures are monitored and controlled by thermocouples: input; intermediate and output (Figure 4). The pyrolysis was carried out in the absence of oxygen and with a frequency inverter set at 1 Hz, which resulted in a time of 30 min between feeding and unloading the material. Temperature control was carried out in the intermediate zone since the collector is located after this zone and before the outlet. The latter corresponds to the gas outlet zone and, as it is the closest zone to the heating point, it presented a temperature higher than the intermediate zone.

Biochar production parameters

In order at produced biochar, different pyrolysis temperatures were used between the ranges of 250 to 420 ºC. The choice of torrefaction/pyrolysis temperatures aimed to evaluate the quality of biochar produced at milder temperatures, which can provide energy savings in the process.

Operational frequency of the electric drive motor screw conveyor was the 1 Hz, which resulted in a total biomass passage time of 30 minutes through the reactor. The pyrolysis temperatures were adjusted in the intermediate zone of the reactor, which totals a time of 10 minutes until the collector, considered as residence time. The temperature in the feed zone was 20 °C and in the inlet zone it was 50 $\pm$ 10% of the pyrolysis temperature (See Figure 3). With these data it was possible to estimate the heating rate, which varied from 9 to 16°C per minute and was considered slow (Table 2).

Figure 1: A and C: *Acacia mearnsii*; B and D: *Eucalyptus dunnii*.

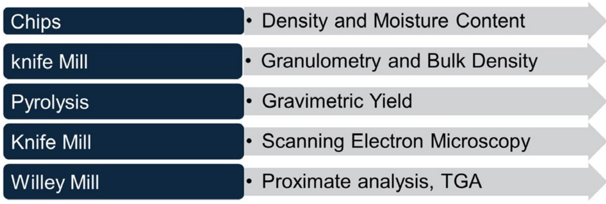

Figure 2: Material preparation steps and analysis.

The pyrolysis of *E. dunnii* wood at a temperature of 420 °C was not carried out because the yield obtained at a temperature of 350 °C was 32%, stipulated a minimum yield to be obtained in biochar. For *A. mearnsii*, pyrolysis was tested at 420 °C, but the obtained yield of 23% demonstrates that this temperature was too high for biochar production under the stipulated conditions.

Characterization of wood and biochar

The energetic properties were determined, which consisted of the proximate chemical analysis and determination of the heating value. This analysis followed the determinations of ASTM D1762 – 84 (2013). The scope of the standard covers the determination of moisture, volatile matter and ash in charcoal. The test method is applicable to chips and briquettes and is designed for the quality assessment of charcoal. To estimate the higher heating value, data from proximate chemical analysis were used, through equation 1, developed by Parikh et al. (2005).

$$HCV = (0{,}3536 * FC + 0{,}1559 * VC - 0{,}0078 * AC) * 238{,}8 \quad (1)$$

Where: HCV = Higher calorific value (Kcal/Kg)
FC = Fixed carbon content (%)
VC = Volatile content (%)
AC = Ash content (%)

For *E. dunnii* and *A. mearnsii* wood, thermogravimetric analysis was performed to compare the results with the gravimetric yield obtained for each wood species in the reactor pyrolysis. The graph was drawn to show the TGA and DTG curves along with the straight lines of the gravimetric yield drawn between the reactor pyrolysis temperature points of 250, 300 and 350 °C for each wood species and 420 °C for *A. mearnsii*.

To obtain the TGA and DTG curves, samples of wood of both species in granulometry between 42 and 65 mesh Tyler were submitted to thermogravimetric analysis in a Shimadzu\TGA50 equipment, at a heating rate of 11 °C/min, in a flow of 50 ml/min of nitrogen.

Surface area, pore volume and pore diameter were characterized by N_2 adsorption at 77 K in a Surface Area and Pore Size Analyzer (Quantachrome Instruments, Nova 1200, USA). Samples were degassed under N_2 flow at 120 °C for 20 h prior to testing. The surface area was determined by 11-points BET method, and pore size distribution by the density functional theory (DFT).

For morphological and chemical analysis of wood and biochar, micrographs were obtained by scanning electron microscopy (SEM) in a MIRA3 TESCAN equipment, with magnifications varying between 50 and 5,000 times and 10 KV of incident beam energy. The samples were covered with a gold layer and glued to individual stainless-steel supports, in the form of small cylinders measuring 1.2 cm in diameter by 1 cm in height.

Figure 3: Pyrolysis reactor - screw conveyor type (Pyrolysis of biomass/waste) and temperature displays.
Note: THERMOCOUPLE/POSITION: E: input; IN: middle; S: output; CO: collector.

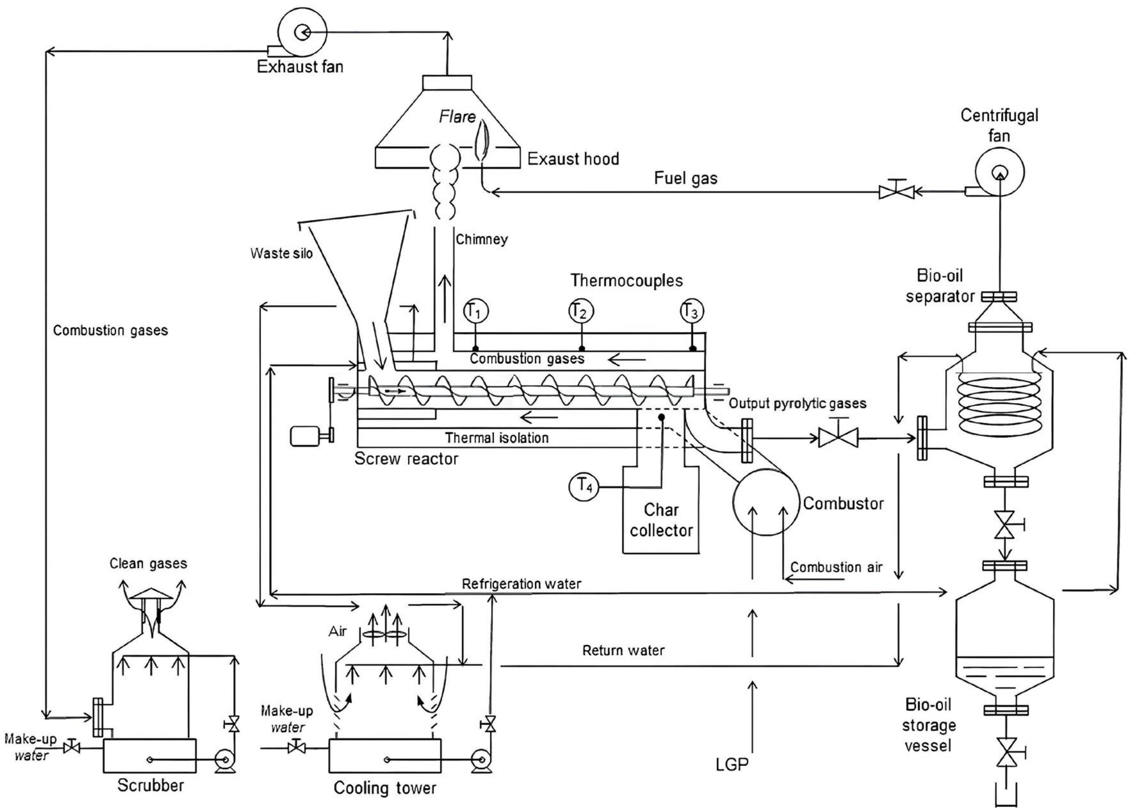

Figure 4: Flowchart of the pyrolysis reactor (Source: Ferreira et al, 2015).

Table 2: Biochar production parameters.

Biochar type	Species	Temperature (°C)	Heating rates (°C / min)	Residence time at temperature (min)
A250	*Acacia mearnsii*	250	9	10
A300	*Acacia mearnsii*	300	11	10
A350	*Acacia mearnsii*	350	13	10
A420	*Acacia mearnsii*	420	16	10
E250	*Eucalyptus dunnii*	250	9	10
E300	*Eucalyptus dunnii*	300	11	10
E350	*Eucalyptus dunnii*	350	13	10

Since the SEM machine is equipped with an Energy Dispersive X-ray Spectrometer (EDS), spectra micrographs of each biomass and biochars at temperatures of 250 to 350 °C were produced. The following elements: C; O; K; Ca; Cl; Mn; Mg; Si were identified and presented in a representative graphical summary. According to Severin (2004), EDS from an SEM sample with minimal preparation can be used to obtain a spectrum easily interpreted by the machine's software, thus obtaining a qualitative elemental analysis.

No preparation of the specimen area subjected to the X-ray on the EDS were made, because in this study the purpose of this analysis was to identify the trend of increase in carbon and decrease in oxygen in the biochar and the possible inorganic elements present in the samples. Although EDS analysis can be used for quantitative determinations, specimen geometry, contaminants and sample preparation associated errors have not been controlled to allow quantitative analysis (Newbury and Ritchie, 2013; Wyroba et al., 2015).

RESULTS

In the energetic properties of the two wood species, it was found that the contents of volatile materials and fixed carbon were similar, as well as the higher calorific value. On the other hand, *E. dunnii* wood presented lower humidity and higher ash content (Table 3).

In a comparison of the reactor pyrolysis yield with the curve obtained by TGA, in the range of temperatures used in this study, it was found that at a temperature of 250 °C the carbonization effect obtained in the reactor followed the trend of the TGA curve. At 350 °C there was greater degradation in the reactor pyrolysis than the mass loss obtained in TGA for the two wood species studied. Before 400 °C, the TGA mass loss stabilized for both species, the same trend followed by the reactor pyrolysis mass loss for *A. mearnsii,* which was the only species pyrolyzed at this temperature. The highest degradation rate of the wood of both species revealed by DTG occurred between 350 and 400 °C. (Figure 5).

In Figure 6 we show the surface area of pores as a function of pyrolysis temperature and pore size distribution of biochars by DFT. In the Table 4 we present the textural properties. An increase in specific surface area is observed with increasing carbonization temperature. Bigger increases were produced at temperatures of 350 °C for *E. dunnii* and 420 °C for *A. mearnsii*, however, with decrease in pore size.

In the microscopic analysis, it appears that there was little change in the morphology of the biochar particles produced at 250 and 300 °C in relation to the wood particles, but at 350 °C there were morphological changes (Figure 7). A graphical representation of the spectra micrographs shows the C; O; K; Ca; Cl; Mn; Mg and Si elements in the woods and the biochar (Figure 8).

DISCUSSION

Energetic properties

In general, the woods of the two species presented similar energetic properties and were close to the averages found by other researchers (Brand et al., 2010; Schwerz et al.,

Table 3: Energy properties of the wood and the biochar produced.

Wood/ *Biochar*	Species	Temp (°C)	GY (%)	MC (%)	VC (%)	FC (%)	AC (%)	HCV (Kcal/Kg)
A000	*A. mearnsii*	----	----	4.64	85.62ab	14.09f	0.29c	4377e
A250	*A. mearnsii*	250	100	5.99	86.06a	13.62f	0.33c	4353e
A300	*A. mearnsii*	300	86	3.72	78.74c	21.38d	0.66b	4706d
A350	*A. mearnsii*	350	39	4.48	52.28d	46.16c	1.56a	5841c
A420	*A. mearnsii*	420	23	6.19	22.58f	75.89a	1.54a	7246a
E000	*E. dunnii*	----	----	3.75	85.42a	14.17f	0.41bc	4376e
E250	*E. dunnii*	250	96	5.69	84.77ab	14.86f	0.36c	4410e
E300	*E. dunnii*	300	85	3.44	82.88b	16.73ef	0.40bc	4497e
E350	*E. dunnii*	350	32	5.84	36.10e	62.48b	1.42a	6617b
Experimental coefficient of variation (%)					3.56	3.78	11.50	3.86

Note: Temp: Temperature; GY: Gravimetric yield; MC: Moisture content; TV: Volatile content; TC: Fixed carbon content; TZ: Ash content; HCV: Higher calorific value. Means followed by the same letter do not differ statistically according to the Tukey test at 95% probability for AC (equal variances) and Dunnett's T3 test at 95% probability for VC, FC and HCV (no equal variances).

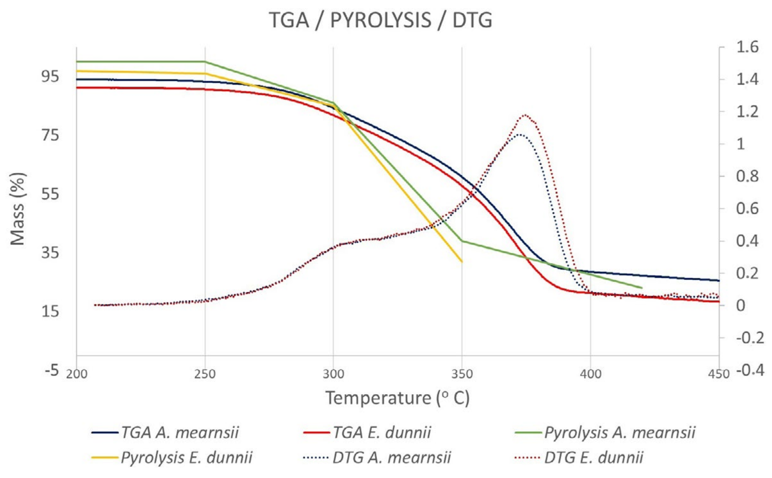

Figure 5: Graphic of the TGA and DTG curves in an inert atmosphere and straight lines of the gravimetric yield drawn between the reactor pyrolysis temperature points of 250, 300 and 350 °C for each wood species and 420 °C for *A. mearnsii*.

2019). The low ash content is highlighted as an advantage, on the other hand the fixed carbon content is low. Temperature of 350 °C promoted an increase in higher calorific value of 33% for *A. mearnsii* and 51% for *E. dunnii*, with gravimetric yield of 39% and 32%, respectively. The pyrolysis of wood at a temperature of 420 °C for *A. mearnsii* wood, contributed to improve the energetic properties of the resulting biochar, but considerably reduced the gravimetric yield.

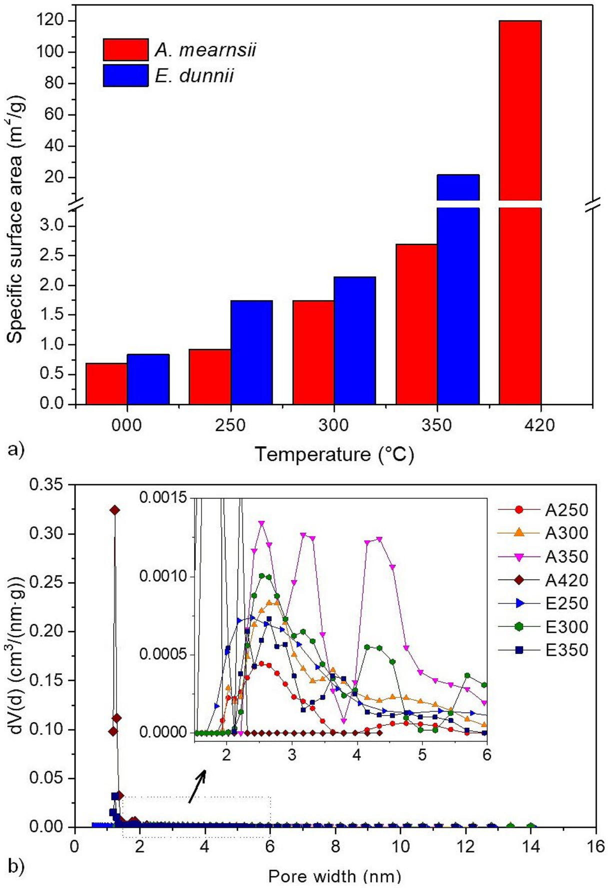

Figure 6: a) Specific surface area of biomass and biochars and b) Pore size distribution of biochars by DFT (inset shows a detail in the region of narrow mesopores).

At the temperatures used, it was found that the 250 and 300 °C was not efficient to carbonize the wood of both species. Biochar has its yield favored, in relation to biogas and bio-oil, by slow pyrolysis with temperatures ranging from 300 to 800 °C (Kan et al., 2016). In the case of this study, however, the temperature of 300 °C produced little carbonization effect, with mass loss of 4 and 5% and increase in fixed carbon content of 7.29 and 2.56 percentage points for *A. mearnsii* and *E. dunnii*, respectively.

Other variables than temperature that influence the yield and properties of the biochar produced and include the residence time of the biomass in the reactor and the type and dimensions of the biomass (Brassard et al., 2017; Kan et al. al., 2016).

Table 4: Textural properties of wood/biochar samples.

Wood/ Biochar	Surface area (m²/g)	Average pore size (nm)	Total pore volume (cm³/g)
	Acacia mearnsii		
A000	0.7	11.1	0.002
A250	0.9	15.8	0.004
A300	1.7	13.5	0.006
A350	2.7	9.1	0.006
A420	120.2	2.5	0.075
	Eucalyptus dunnii		
E000	0.8	8.0	0.002
E250	1.7	6.6	0.003
E300	2.1	6.7	0.004
E350	21.9	2.9	0.016

In the analysis of the gravimetric yield and volatile content of Table 3, together with Figure 5, *E. dunnii* wood showed greater degradation at temperatures of 250 and 300 °C, but with a small portion of loss in the volatile content of the resulting product. For *A. mearnsii* wood at a temperature of 250 °C there was no degradation and at 300 °C the degradation was close to that observed with *E. dunnii*.

In a vertical reactor using an inert atmosphere with a flow of 500 ml/min of nitrogen, temperature of 400 °C, residence time of 30 min and heating rate of 5 °C/min, Hillig et al. (2020) obtained biochar from pine wood with 70% fixed carbon and 1.61% ash content.

Pyrolysis line and TGA/DTG analysis

Difference between the pyrolysis line and the TGA line was attributed to TGA analysis occurred at controlled atmosphere while in the reactor at the restricted atmosphere. Furthermore, the pyrolysis temperature was adjusted in the intermediate zone (see Figure 3) and a passage time of 10 minutes for the wood was estimated between this zone and the collector.

The DTG curves of both species were similar, with the highest degradation peak between the temperatures of 350 and 400 °C. These peaks, however, started at temperatures below 350 °C, which confirms the effect of the residence time of the material in the reactor on pyrolysis.

The smaller biomass particle size visually observed for *E. dunnii* may have contributed to its lower biochar yield. According to Kan et al. (2016) the smaller particle size favored the formation of bio-oil to the detriment of the biochar formation.

Yu et al. (2018) using a fixed bed reactor, found that increasing the biomass particle size obtained from Beechwood promoted an increase in biochar yield, but especially at temperatures below 400 °C. Furthermore, the TGA and DTG curves demonstrate a more accentuated degradation for *E. dunnii* than the *A. mearnsii* (Figure 5).

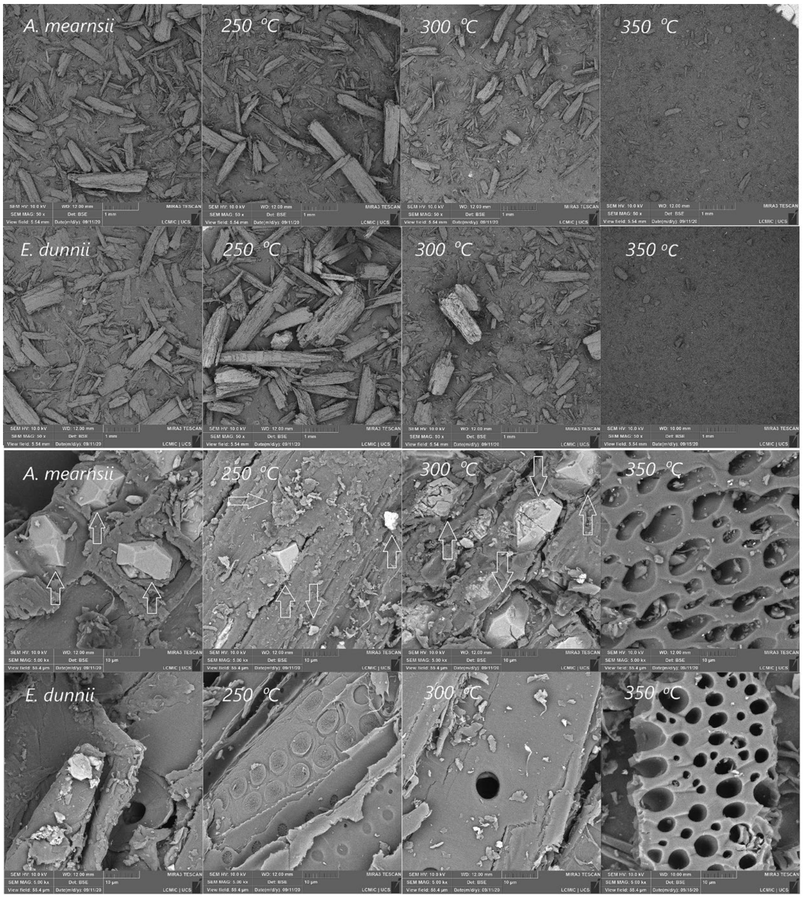

Figure 7: Micrographs of wood and biochar produced at different temperatures. 1st and 2nd row: 50x; 3rd and 4th row: 5,000x. Arrows in the third row indicate cellulose micro-crystals in the *A. mearnsii* particles, which suffered little degradation in the pyrolysis at 250 and 300 °C.

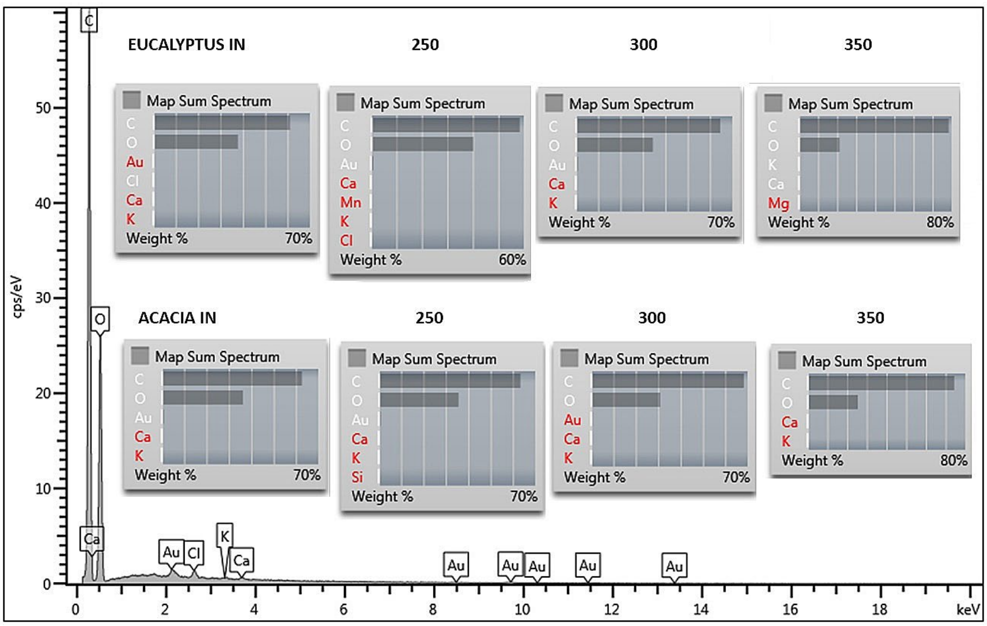

Figure 8: Summary representation of spectra micrographs obtained from Energy Dispersive X-ray Spectrometer (EDS) of the woods and the biochars at different temperatures.

The TGA and DTG curves obtained in the thermogravimetric analysis for both species was similar, with three degradation peaks more accentuated for *E. dunnii*, which correspond to the loss of extractives (below 100 ºC), loss of hemicelluloses (slightly above 300 ºC) and loss of cellulose (close to 380 ºC). The degradation of lignin occurs between 250 and 600 ºC and therefore did not show a characteristic degradation stage (Carrillo et al. 2018), thus, the more pronounced degradation of *E. dunnii* above 250 ºC was attributed to its higher proportion of lignin determined for the *A. mearnsii* and *E. dunnii* woods in other studies (Hodge et al., 2018; Marinho et al., 2017).

Thus, in this study, *E. dunnii* wood showed lower biochar yield than *A. mearnsii* wood because its lower density and smaller particle size in combination with its chemical characteristics had an influence on pyrolysis.

Morphological aspects of biochar

Increase in surface area with increasing carbonization temperature is related to the greater release of volatile matter provided by higher temperatures, as discussed earlier. Granados et al. (2017) also obtained biochar with surface area in this range (0.5 - 2.1 m^2/g) in the thermochemical processing of Poplar wood in an inclined rotary torrefier at 280 and 300 ºC and different residence times.

The greater susceptibility of *E. dunnii* to degradation observed both in the yields obtained in the pilot reactor and in the degradation curve by thermogravimetric analysis, was also reflected in the development of the surface area. It is observed that for all temperatures studied, *E. dunnii* biochars have a larger surface of pore area when compared to *A. mearnsii* biochars.

The higher specific surface area observed for the *E. dunnii* is due to the smaller size of the pores formed during carbonization. Figure 6b also makes it clear that, for the same temperature condition, there is a greater development of pores in the micropores/narrow mesopores region for the *E. dunnii* wood samples.

Micropore formation is more pronounced at elevated temperatures, resulting in a material with the largest surface area at a temperature of 350 ºC for *E. dunnii* (21,9 m^2/g) and 420 ºC for *A. mearnsii* (120,2 m^2/g). It is known that the increase in surface area is related to the increase in the total volume of pores and to the decrease in the average pore size (Cao et al. 2015).

In this case, for more noble applications of biochar such as soil conditioning or activated carbon, temperatures between 350 and 420 ºC can be tested for both species. The best relationship between gravimetric yield and porosity can be sought for each case.

In the micrographs at 5,000x magnification, it is possible to observe cellulose micro-crystals in the *A. mearnsii* particles, which suffered little degradation in the pyrolysis at 250 and 300 ºC. At the 350 ºC, these micro-crystals were degraded resulting in the typical porous structure of charcoal. For *E. dunnii* it was not possible to observe the cellulose as clearly, however, the typical porous structure is very clear in the biochar produced at 350 ºC.

The biochar structure of both woods presented pores of different dimensions, in the same way as Banczek et al. (2019) verified in charcoal obtained from the *Pinus elliottii* wood. The authors evaluated the morphological characteristics of the charcoal with and without activation, verifying an increase in the charcoal porosity after the activation process, mainly due to the increase in micro-pores.

Finally, the EDS of the micrographs showed the presence of traces (less than 2%) of inorganic elements present in the wood and biochar samples, the main ones being calcium and potassium. It was also possible to verify that at temperatures up to 300 ºC there was little variation in carbon and oxygen in its chemical composition, which changes more markedly at a temperature of 350 ºC and for *E. dunnii* wood.

CONCLUSIONS

In this study, the properties of Biochar produced with *Eucalyptus dunnii* and *Acacia mearnsii* woods in a horizontal reactor were evaluated to verify the effect of species and torrefaction/pyrolysis temperature on its energy properties.

The use of temperature up to 300 ºC produced little pyrolysis effect, being that at 350 ºC it was pronounced and at 420 ºC it was considered excessive because it resulted in a low yield, for *A. mearnsii* wood of 23%.

E. dunnii wood showed lower biochar yield at the temperatures used, a fact attributed to its greater degradation in thermogravimetric analyses, its lower density and the smaller particle size obtained in its processing.

The temperature of 350 ºC was considered as the most suitable for the biochar production for the energy purposes, from both wood species in this type of reactor, with a residence time of 30 min.

Eucalyptus dunnii presented the higher specific surface area than *Acacia mearnsii* in temperature of the 350 ºC, but this species had a considerable increase in surface area at a temperature of 420 ºC. Thus, for the soil conditioning purposes, is suggested to test the appropriate temperatures for each species, between 350 and 420 ºC.

AUTHORSHIP CONTRIBUTION

Project Idea: EH; MG

Funding: EH; MG

Database: EH; DP; CM; OAN

Processing: EH; DP; CM; OAN

Analysis: EH; DP; CM; OAN

Writing: EH; CM

Review: EH; MG; DP; CM

ACKNOWLEDGEMENTS

The authors are grateful to the companies and institutions that collaborated with this project: Midwestern Parana State University, UNICENTRO; University of Caxias do Sul, UCS; SETA S/A and KLABIN S/A.

REFERENCES

American Society for Testing and Materials - ASTM. (2013). D1762-84 Standard Test Method for Chemical Analysis of Wood Charcoal. ASTM International, West Conshohocken. http://www.astm.org/Standards/D1762.htm

BANCZEK, E. P.; BRUGNERA, A. B.; CHRISTOFORO, A. L.; et al. Morphological and elementary evaluation of wooden carbonaceous materials from activated carbon industry. Nativa, v.7, n.2, p.213-217, 2019.

BASU, P. Biomass gasification, pyrolysis and torrefaction: Practical design and theory. Elsevier, 2018. 564p.

BENTANCOR, L.; HERNÁNDEZ, J.; DEL PINO, A.; et al. Evaluation of the biomass production, energy yield and nutrient removal of *Eucalyptus dunnii* Maiden grown in short rotation coppice under two initial planting densities and harvest systems. Biomass and Bioenergy, v.122, p.165-174, 2019.

BRAND, M. A.; MUÑIZ, G. I. B. D.; QUIRINO, W. F.; et al. Influence of storage time on the quality of biomass for energy production in humid subtropical regions. Cerne, v. 16, n.4, p.531-537, 2010.

BRASSARD, P.; GODBOUT, S.; RAGHAVAN, V. Pyrolysis in auger reactors for biochar and bio-oil production: A review. Biosystems engineering, v.161, p.80-92, 2017.

CAO, T.; SONG, Z.; WANG, S.; et al. A comparative study of the specific surface area and pore structure of different shales and their kerogens. Science China Earth Sciences, v.58, p.510-522, 2015.

CARRILLO, I.; MENDONÇA, R. T.; AGO, M.; et al. Comparative study of cellulosic components isolated from different Eucalyptus species. Cellulose, v.25, n.2, p.1011-1029, 2018.

CHAN, J. M.; DAY, P.; FEELY, J.; et al. *Acacia mearnsii* industry overview: current status, key research and development issues. Southern Forests: a Journal of Forest Science, v.77, n.1, p.19-30, 2015.

CIUTA, S.; TSIAMIS, D.; CASTALDI, M.J. Chapter Two - Fundamentals of Gasification and Pyrolysis, In: Gasification of Waste Materials, Academic Press, p. 13-36, 2018.

DIAS JUNIOR, A. F.; ESTEVES, R. P.; SILVA, A. M.; et al. Investigating the pyrolysis temperature to define the use of charcoal. European journal of wood and wood products, v.78, n.1, p.193-204, 2020.

DOBNER JR, M.; BATISTA, K. M.; SARTÓRIO, I. P.; et al. Crescimento e desempenho econômico de *Eucalyptus dunnii* em diferentes sítios no Planalto Sul do Brasil (Growth and economic performance of *Eucalyptus dunnii* at different sites on the highlands of southern Brazil). Floresta, v.47, n.4, p. 397-406, 2017.

DUFOURNY, A.; VAN DE STEENE, L.; HUMBERT, G.; et al. Influence of pyrolysis conditions and the nature of the wood on the quality of charcoal as a reducing agent. Journal of Analytical and Applied Pyrolysis, v.137, p.1-13, 2019.

FERREIRA, G. W.; OLIVEIRA, F. C.; SILVA, L. O.; et al. Nitrogen alters initial growth, fine-root biomass and soil organic matter properties of a *Eucalyptus dunnii* Maiden plantation in a recently afforested grassland in Southern Brazil. Forests, v.9, n.62, 2018.

FERREIRA, S. D.; ALTAFINI, C. R.; PERONDI, D.; et al. Pyrolysis of Medium Density Fiberboard (MDF) wastes in a screw reactor. Energy Conversion and Management, v. 92, p.223-233, 2015.

GIESBRECHT, B. M.; COLDEBELLA, R.; GENTIL, M.; et al. The performance of *Acacia mearnsii* De Wild for kraft pulping. Ciência Florestal, v.32, n.1, p.266-286, 2022.

GRANADOS, D. A.; BASU, P.; CHEJNE, F.; et al. Detailed investigation into torrefaction of wood in a two-stage inclined rotary torrefier. Energy & Fuels, v.31, n.1, p.647-658, 2017.

GRIFFIN, A. R.; MIDGLEY, S. J.; BUSH, D.; et al. Global uses of Australian acacias recent trends and future prospects. Diversity and Distributions, v.17, n.5, p.837-847, 2011.

HILLIG, D. M.; POHLMANN, J. G.; MANERA, C.; et al. Evaluation of the structural changes of a char produced by slow pyrolysis of biomass and of a high-ash coal during its combustion and their role in the reactivity and flue gas emissions. Energy, v.202, p. e117793, 2020.

HODGE, G. R.; ACOSTA, J. J.; UNDA, F.; et al. Global near infrared spectroscopy models to predict wood chemical properties of Eucalyptus. Journal of Near Infrared Spectroscopy, v. 26, n. 2, p. 117-132, 2018.

HUANG, Y.; LI, B.; LIU, D.; et al. Fundamental advances in biomass autothermal/oxidative pyrolysis: a review. ACS Sustainable Chemistry & Engineering, v.8, n.32, p.11888-11905, 2020.

JOVANOVIC, T.; ARNOLD, R.; BOOTH, T. Determining the climatic suitability of *Eucalyptus dunnii* for plantations in Australia, China and Central and South America. New Forests, v.19, n.3, p.215-226, 2000.

KAN, T.; STREZOV, V.; EVANS, T. J. Lignocellulosic biomass pyrolysis: A review of product properties and effects of pyrolysis parameters. Renewable and Sustainable Energy Reviews, v.57, p.1126-1140, 2016.

KLABIN. Resumo Público - Plano de manejo florestal - Santa Catarina, 2024. Available at: https://klabin.com.br/sustentabilidade/estrategia/resumo-publico. Accessed in: May 15th 2024.

LUDVICHAK, A. A.; SCHUMACHER, M. V.; VIERA, M.; et al. R. Growth, biomass and nutrient stock in mixed-species planting of hybrid *Eucalyptus urograndis* and *Acacia mearnsii* in Southern Brazil. New Forests, v.53, p.203-219, 2022.

MARINHO, N. P.; KLOCK, U.; LENGOWSKI, E. C.; et al. Características da Polpa kraft Extraída da Espécie Acácia-negra na Produção de Papel (Characteristics of the Kraft Pulp Extracted from Black-Wattle Species in Papermaking). Floresta e Ambiente, v.24, p.e00099214, 2017.

MERMOUD, F.; SALVADOR, S.; VAN DE STEENE, L.; et al. Influence of the pyrolysis heating rate on the steam gasification rate of large wood char particles. Fuel, v.85, n. 10-11, p. 1473-1482, 2006.

NEWBURY, D. E.; RITCHIE, N. W. M. Is scanning electron microscopy/energy dispersive X-ray spectrometry (SEM/EDS) quantitative? Scanning, v.35, n.3, p.141-168, 2013.

PARIKH, J.; CHANNIWALA, S. A.; GHOSAL, G. K. A correlation for calculating HHV from proximate analysis of solid fuels. Fuel, v.84, n.5, p.487-494, 2005.

PEREIRA, M. P. D. C. F.; COSTA, E. V. S.; PEREIRA, B. L. C.; et al. Torrefação de cavacos de eucalipto para fins energéticos (Torrefaction of eucalyptus wood chips for energy purposes). Pesquisa Florestal Brasileira, v.36, n.87, p.269-275, 2016.

RESQUIN, F.; NAVARRO-CERRILLO, R. M.; CARRASCO-LETELIER, L.; et al. Influence of contrasting stocking densities on the dynamics of above-ground biomass and wood density of *Eucalyptus benthamii, Eucalyptus dunnii,* and *Eucalyptus grandis* for bioenergy in Uruguay. For. Ecol. Manag, v.438, p.63-74, 2019.

RESQUIN, F.; NAVARRO-CERRILLO, R. M.; CARRASCO-LETELIER, L.; et al. Influence of age and planting density on the energy content of *Eucalyptus benthamii, Eucalyptus dunnii* and *Eucalyptus grandis* planted in Uruguay. New Forests, v.51, n.4, p.631-655, 2020.

RODRIGUES, T. O.; ROUSSET, P. L. A. Effects of torrefaction on energy properties of *Eucalyptus grandis* wood. Cerne, v.15, n.4, p.446-452, 2015.

SCHWERZ, F.; ELOY, E.; ELLI, E. F.; et al. Reduced planting spacing increase radiation use efficiency and biomass for energy in black wattle plantations: Towards sustainable production systems. Biomass and Bioenergy, v.120, p.229-239, 2019.

SETA. Resumo Público - Plano de Manejo Florestal, 2022. Available in: https://www.setaoficial.com/files/downloads/resumo-publico-plano-de-manejo-florestal-2022-web.pdf. Accessed in: May 15th 2024.

SEVERIN, K. P. Energy Dispersive Spectrometry. In: Energy Dispersive Spectrometry of Common Rock Forming Minerals. Springer, Dordrecht, p 1-13, 2004.

SOMERVILLE, M.; DEEV, A. The effect of heating rate, particle size and gas flow on the yield of charcoal during the pyrolysis of radiata pine wood. Renewable Energy, v. 151, n.8, p.419-425, 2020.

TANCREDI, N.; CUÑA, A.; YOSHIDA, M. I. Wood Pyrolysis: Influence of Pyrolysis Temperature and Heating Rate on Charcoal Properties and Pyrolysis Process. Chapter: 12. In: LAPHERTY, J. N. New Trends in Chemical Physics Research. New York: New Science Publishers, inc., p. 225-236, 2011.

TRIPATHI, MANOJ; SAHU, JAYA NARAYAN; GANESAN, P. Effect of process parameters on production of biochar from biomass waste through pyrolysis: a review. Renewable and Sustainable Energy Reviews, v.55, p.467-481, 2016.

WYROBA, E.; SUSKI, S.; MILLER, K.; et al. Biomedical and agricultural applications of energy dispersive X-ray spectroscopy in electron microscopy. Cellular and Molecular Biology Letters, v.20, n.3, p.488-509, 2015.

YU, J.; SUN, L.; BERRUECO, C.; et al. Influence of temperature and particle size on structural characteristics of chars from Beechwood pyrolysis. Journal of Analytical and Applied Pyrolysis, v.130, p.127-134, 2018.