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Pyrogenic Carbonaceous Materials Production of Four Tropical Wood Produced by Slow Pyrolysis at Different Temperatures: Charcoal and Biochar Properties

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Abstract: Costa Rica produces a considerable, important quantity of wood residues. This waste can be pyrolyzed to produce charcoals as main products that can be effectively used as an energy source or to immobilize carbon for soil treatment. However, there is a lack of information about the pyrogenic carbonaceous materials (PCMs), such as charcoal or biochar, obtained at different pyrolysis temperatures. Hence, this study aimed to evaluate the quality of PCMs (physical, mechanical, ultimate analysis, and FTIR analysis) and charcoal characteristics (energetic properties and thermogravimetric analysis—TGA) and biochar characteristics (conductivity, pH, initial contact angle, and wetting rates) for four tropical wood residues produced in five temperatures (300 °C, 350 °C, 400 °C, 450 °C, and 500 °C). In general, pyrolysis temperature between 450 °C and 500 °C produced charcoals with lower values of density, moisture content, compression strength, volatiles, H and O content, and higher values of C and ash contents, conductivity, pH, initial contact angle, and wetting rates. FTIR and TGA analyses show that celluloses and lignin are pyrolyzed at these temperatures, so these temperatures are recommended. The range of 300–350 °C is not recommended, as these parameters were inverse. Multivariate analysis shows that (i) PCMs obtained at lower temperatures (300–350 °C) from *Dipteryx panamensis*, *Hieronyma alchorneoides*, and *Tectona grandis* belong to a cluster with poorer properties, indicating that these temperatures are not adequate for pyrolysis of these species; (ii) all the PCMs obtained from *Gmelina arborea* were grouped into one cluster, suggesting different PCM quality; and (iii) the PCMs produced from *D. panamensis*, *H. alchorneoides*, and *T. grandis* at 400–500 °C were grouped into another cluster with better properties, suggesting this pyrolysis temperature range as the best for these species.

Keywords: carbonization; tropical wood; pyrolysis liquids; biochar



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1. Introduction

Pyrolysis is a thermochemical process that is applied to biomass, in which the main matrix polymers (cellulose, hemicellulose, and lignin) are decomposed in non-oxidizing atmospheres. This process results in solid product (charcoal or/and biochar), two types of liquid, which can be separated in bio-oil or wood vinegar, and non-condensable gases [1] with a variety of uses, including charcoal as fuel (a more traditional use), bio-oil as a soil amendment (biochar), activated carbon, chemical products, and many other uses [2]. Specially, the solid product is pyrogenic carbonaceous material (PCM), produced by thermochemical conversion of carbonaceous feedstock. If the PCM is used as a fuel, i.e., it is burned and the carbon is transformed (oxidized) into CO₂, it is classified as charcoal, but if the PCM is used for agricultural purposes or the application is a multifunctional material that can be used to address several challenges within a life cycle, the solid product

of pyrolysis is called biochar [3]. Thus, in this article the term charcoal is used to refer to a fuel purpose and biochar for a soil purpose.

The pyrolysis process is carried out in a closed chamber (reactor) in the absence of oxygen and biomass decomposition process is carried out through different mechanisms that include dehydration, depolymerization, isomerization, aromatization, decarboxylation, and charring of cellulose, lignin, and hemicellulose [4,5]. Cellulose, the glycosidic bonds linking in glucose units, are not strong and break down at high temperatures and the products of cellulose pyrolysis are acids, alcohols, anhydrous sugars, charcoal, and gases, but furans and laevoglucose can also be formed by other mechanisms in the cleavage of $\beta - 1, 4$ -glycosidic bonds [6]. In the case of hemicellulose, it occurs at low temperatures with high CO_2 production and high charcoal or biochar production [7]. Lignin is a more complex polymer; almost all kinds of structural units obtained during degradation are formed by oxidation of noniferyl alcohol, synapyl alcohol, and *p*-coumaryl alcohol [8].

The thermal degradation of lignocellulosic biomass with temperature in the absence of oxidants is currently classified into three types according to the heat rate, temperature, and residence of solids in the reactor: slow pyrolysis, fast pyrolysis, and flash pyrolysis [9]. Slow pyrolysis occurs at relatively low temperatures ($<500\text{ }^\circ\text{C}$) with a long residence time, is generally called carbonization, and is widely used for PCM production in addition to maximizing this solid product. The production of the liquid or condensable phase (wood vinegar) is maximized and the production of bio-oil is low [10]. Temperature and heating rate play an important role in the quality of biochar. In this type of pyrolysis, the low heating rate reduces secondary pyrolysis and the thermal cracking process in both cellulosic polymers (cellulose and hemi-cellulose) and lignin, resulting in PCM as the main product [11].

Slow pyrolysis is a process that occurs in areas with little technological development in pyrolysis equipment, and is perhaps the best known and most popular method in regions such as the developing countries of the tropical areas of the Americas [12]. Costa Rica, a small country in Central America, has this condition of little development in pyrolyzer equipment, because its energy matrix is based on the production of hydroelectric energy, so other energy sources such as biomass are less important, even though there is significant availability [13]. In addition to little development in equipment, the country has a large amount of productive activities that are carried out by very important agricultural crops, meaning a great demand for fertilizers and agrochemicals, which are currently not considered as a good sustainability policy for the agricultural sector [14,15]. Thus, the agriculture sector is increasingly looking for more appropriate options for crop management, which include soil management and developing more environmentally friendly agrochemicals [15,16].

The effect of temperature in slow pyrolysis on the yield of PCM and the different pyrolysis products and characteristics in various types of biomasses has been studied [17,18]. However, optimization of the temperature and the pyrolysis process itself is complex as it depends on the type of feedstock, composition, physicochemical properties, nature of the application, and other operational factors [18]. Specifically, studies on the production of different pyrolysis products and their behavior in the slow pyrolysis process at different temperatures have been limited to biomass from forest plantations [19].

On the other hand, in Costa Rica forest plantations supply about 78% of the wood market. The yield of marketable sawn wood from these processes is estimated at around 25% of the volume of the forest plantation, evidencing inefficient production processes and a high percentage of residues [20]. Therefore, in the renewal of the wood industry, greater importance must be given to the valorization and use of the residues from these processes, which are useful in the production and cogeneration of electrical and heat energy, production of charcoal and firewood, and organic compost, among others [21]. Therefore, the residues from these plantations can be pyrolyzed, to produce charcoal or biochar, wood vinegar, bio-oil, and non-condensable gases. In the case of biochar, the production can vary

by 25–40% in slow pyrolysis [22] and is currently used in bioenergy, carbon activity, or to manage soil sustainability [23].

Main species planted in Costa Rica are *Dipteryx panamensis*, *Hieronyma alchorneoides*, and *Tectona grandis* [20,21]. The *H. alchorneoides* and *D. panamensis* species produce lumber with high density and their wood is used in structural productions [19]. In the case of *G. arborea* wood, it is used for pallet and furniture fabrication, and *Tectona grandis* is exported for international markets, flooring, and furniture fabrication. The yield of marketable sawn wood from primary sawmills is estimated to be around 25% of the volume of the forest plantation (approx. 187,500 tons per year⁻¹), highlighting inefficient production processes and a high percentage of residues, approx. 562,500 tons per year⁻¹ [20]. Therefore, the renewal of the wood industry calls for increased attention to be paid to the valorization and utilization of the residues generated from these processes, which are useful in the production and cogeneration of electrical and heat energy, production of charcoal and firewood, organic compost, and other applications [21,24]. The pyrolysis of residues from these plantations can produce biochar, wood vinegar, bio-oil, and non-condensable gases [24].

Some studies evaluated the charcoal and biochar qualities of some Costa Rican tropical woods, including energy densities, energy yields, biochar characteristics, and thermal profiles, in addition to other properties [20,24–26], but when specifically studying the Costa Rican species in slow pyrolysis, *Tectona grandis* and *Gmelina arborea* lacked the same attention [27]; however, it does not refer to the effects of temperature in slow pyrolysis [28–31]. A study carried out on *T. grandis* by Parthasarathy and Sheeba [32] studied the yield, solid residence time, and charcoal characteristics produced at different temperatures of slow pyrolysis (300 °C, 350 °C, 450 °C, 500 °C, and 550 °C) and demonstrated that the yield of charcoal decreased, and residence time remained stable. In relation to *G. arborea* studies, it was reported that charcoal characteristics pyrolysis was carried out in a fabricated fixed bed pilot-scale reactor using sawdust biomass to produce bio-oil, but again, they presented the charcoal characteristics [33].

Considering that *Dipteryx panamensis*, *Gmelina arborea*, *Hieronyma alchorneoides*, and *Tectona grandis* are four species of commercial importance in reforestation programs, a significant quantity of residues is produced during harvesting and sawing and can be pyrolyzed to produce charcoal. However, there is a lack of information on charcoal properties obtained from slow pyrolysis. Therefore, the objective of the present study was to determine of PCM properties (physical and mechanical, ultimate, and chemical change by FTIR) and evaluate charcoal characteristics (energetic properties and thermogravimetric analysis—TGA) and biochar characteristics (conductivity, pH, initial contact angle, and wetting rates) of four wood species (*D. panamensis*, *G. arborea*, *H. alchorneoides*, and *T. grandis*) produced in a pilot-scale reactor at different temperatures (300 °C, 350 °C, 400 °C, 450 °C, and 500 °C).

2. Materials and Methods

2.1. Materials

Parent woods from four different woods (*D. panamensis*, *G. arborea*, *H. alchorneoides*, and *T. grandis*) were tested and they came from residues. *D. panamensis* and *H. alchorneoides* residues came from lumber that was dried in drying kiln, and presented a moisture content (MC) of 12% [34,35]. Lumber of *H. alchorneoides* was from a commercial plantation of 12 years of age and lumber of *D. panamensis* from a commercial plantation of 16 years of age. The information about plantation conditions of these two wood species are detailed in Moya and Tenorio [34]. *G. arborea* and *T. grandis* wood was collected from a plantation of 8 years of age [35]. Basal logs from three trees (a diameter breast height of approximately 22 cm) from each plantation were sawn and non-commercial boards with heartwood were selected and 168 specimens of 14.0 cm × 2.5 cm × 2.0 cm (length × width × thickness) were extracted. After this, they were dried until reaching 12% MC. Subsequently, six groups of 28 samples from 160 species were separated: five groups used for PCM production with

five different temperatures and one group for parent wood. The chemical and physical characteristic of parent wood can be consulted in Moya et al. [36].

2.2. Process of Carbonization and Treatment

Five target temperature were tested in each wood residue: 300 °C, 350 °C, 400 °C, 450 °C, and 500 °C. Pilot cylindrical reactor with a capacity of 3 L (12 cm in diameter and 30 cm in length) was used, according to that described by Balaguer-Benlliure et al. [19]. The reactor accommodated 14 pieces of sample wood; two batches were pyrolyzed for each temperature. The temperature was continuously measured at two points with probes: (i) inside of reactor and (ii) in gas outlet of reactor. Testo data logger, model 176/T4 (Testo SE & Co., Titisee-Neustadt, Germany), was used for recording of temperature.

After placing the 14 samples in the reactor, it was sealed and pressurized with nitrogen gas to 2 MPa, then depressurized, to create an oxygen-poor environment. Then, the reactor was heated to 12.5 °C/min until reaching 275 °C and 1.55 MPa, at which moment a valve was opened to slowly release pyrolysis gases. The target temperature was reached and maintained until no more gases were expelled. The reactor was cooled to ambient temperature. The residence time of the pyrolysis varied from 150 to 300 min according to target temperatures and this information was in Moya et al. [24].

2.3. Evaluations and Properties of Pyrogenic Carbonaceous Materials (PCMs)

The evaluation of PCMs included yield, physical properties, ultimate analysis, and chemical changes by FTIR. The properties and parameters evaluated were grouped because they are important in determining charcoal or biochar purpose.

2.3.1. Evaluation of Pyrogenic Carbonaceous Materials (PCMs) Yield

The yield of PCM was calculated according to the procedures outlined by Balaguer-Benlliure et al. [19]. Parent wood was weighed before the pyrolysis process at each temperature and PCM was weighed after cooling stage. PCM yield (charcoal or biochar) was calculated according to Equation (1).

$$\text{Pyrogenic carbonaceous materials yield (\%)} = \frac{\text{Charcol or Biochar (kg)}}{\text{Parentwod weighth (kg)}} * 100 \quad (1)$$

2.3.2. Physical and Mechanical Properties

PCM characteristics determined were density, MC, and compression resistance. A total of 20 charcoal specimens of 2.5 cm × 2.5 cm × 2.0 cm were extracted per temperature for density determination. After, their dimensions were measured for volume determination and were weighed too, allowing for the density to be calculated. The 20 charcoal specimens were dried until reaching constant weight in an oven at 103 °C. MC of these samples was calculated according to American Standard Testing Material (ASTM) D1762-84 Standard [37]. Compression resistance was determined in grain-parallel direction. The samples presented a dimension of 2.0 × 2.0 × 5.cm (width × thickness × length), and were tested at 0.3 mm/min load speed until rupture of sample. Compression resistance was determined in the parent woods as well using universal testing machine (Tinius Olsen, 10 kN model, Horsham, PA, USA).

2.3.3. Ultimate Analysis

Quintuplicate determination of content of C, N, H, and S was determined in 5 mg per each temperature and parent wood. These elements were measured using Elementar Analysensysteme, model Vario Macro Cube (Muchen, Germany). The determination of O content was calculated by subtracting the sum of C, H, and N content from 100. Additionally, the ratios of C/N and H/C and molar ratios of O/C and H/C were calculated [38]. Five samples per temperature/species were used for this determination.

2.3.4. FTIR Analysis

Fourier-transform infrared spectroscopy analysis (FTIR) by means of attenuated total reflection (ATR) was carried out on three samples per pyrolysis temperature and parent wood. The materials were ground and the fraction between 40 and 60 mesh selected, after drying at 105 °C to constant weight. A FTIR spectrometer (a Nicolet 380 FTIR spectrometer Thermo Scientific, Mundelein, IL, USA) was used and configured for 32 exploration readings at 1 cm⁻¹ resolution with background correction. The data were computed with Spotlight 1.5.1, HyperView 3.2, and Spectrum 6.2.0 software, developed by Perkin Elmer Inc. (Waltham, MA, USA). The vibrational bands were selected according to different studies on charcoal [39–43], as described in the following list:

- 2910 is C-H (stretching) and represents loss of hydroxyl bonds by heating;
- 2160–2170 represents CO formation due to carbonization;
- 1700 is C=O bond of hemicellulose and cellulose degradation;
- 1600 and 1630 is C=O (stretching) of the aromatic ring of lignin;
- 1434 is CH₂ and CH₃ (asymmetric charact) of aromatic nuclei in lignin, kept stable;
- 1206 is C-O (stretching) of C-O breakage;
- 1032 is C-O (stretching) and represents the bonds in acids, alcohols, phenols, ether, or ester groups;
- 1111 is C-O (stretching) and represents the bonds in acids, alcohols, phenols, ether, or ester groups;
- 900 is C-H of the out-of-plane of the aromatic ring;
- 878 is C-H of the out-of-plane glucose ring in cellulose and hemicellulose and for guaiacyl ring in lignin;
- 810 and 750 is C-H of the out-of-plane of the aromatic rings.

2.4. Charcoal Characterization

Characterization of the charcoals included energetic properties and thermogravimetric analysis (TGA). In addition to these, some properties associated with uses for soil amendment were assessed.

2.4.1. Energetic Properties

Energy characteristics measured were gross caloric value (GVC), ash, and volatile content. GCV was determined at 0% of moisture content according to ASTM D5865-19 standard and using Parr's calorimetric test [44]; for each material, ten samples of 300 mg per pyrolysis temperature were tested. Ash content was determined in three samples (2 g each) per material of each temperature/species, according to ASTM D3173 standard [37,45]. Three samples weighing 3 g each per temperature/species were used following the ASTM D1762 [37].

2.4.2. TGA Analysis

Thermogravimetric analyzer (TA Instruments Q500, New Castle, DE, USA) was used for TGA analysis. An inert atmosphere was provided by ultra-high purity nitrogen with flow rates of 90.0 mL min⁻¹. One sample of 5 mg of charcoal were used for each temperature and parent wood. Each analysis was developed beginning with a thermal stabilization and isothermal period at 30 °C and 10 min. The heat rate was 25 °C/min until 750 °C. TA Instruments Universal Analysis 2000 software was used in data acquired.

2.5. Biochar Characteristics

The charcoal was analyzed considering four parameters evaluated for agricultural applications according to Masís-Meléndez et al. [46]: electrical conductivity, pH, hydrophobicity using the initial contact angle, and wettability using wetting rate. For electrical conductivity and pH, charcoal was milled until a size of 420 µm and 250 µm, 40 and 60 mesh, respectively, and four samples of 2 g were taken temperature/species. For sample preparation, the sample was added in a 50 mL plastic flask with 30 mL of water and the

mix was shaken in an orbital shaker (Hotech Instruments Corp. 720R, TPE, Taipei, Taiwan) for 1 h at 30 rpm and 25 °C. Finally, the sample was filtered.

Firstly, the pH of the filtered solution was measured using a benchtop meter (Oakton, ION 700, Environmental Express, Charleston, SC, USA). The conductivity of the solution was measured with Hanna Instruments, model HI99312 (Woonsocket, RI, USA). The sample of biochar was spread out on a double-sided adhesive tape fastened at the end of a microscope glass slide (24.4 mm × 76.2 mm) for hydrophobicity properties. Particles of biochar (size fraction < 63 µm) were pressed with a weight of 100 g onto the tape for 5 s. After, a droplet of 2 µL of water was placed on a particle of biochar and the contact angle was measured, according to Batchmann et al. [47]. The initial contact angle (θ_{initial}) was recorded every 10 s for 1200 s (20 min), using goniometer ramé-hart Model 590 (Ramé-hart instruments Co., Succasunna, NJ, USA) with DROPimage CA software (Finn Knut Hansen, OS, Norway) The wettability was determined by two contact angles measured: θ_{initial} and the contact angle at 20 min (θ_{20}). The wetting rate was calculated as the variation in the contact angle ($\theta_{20} - \theta_{\text{initial}}$) over 20 min of wetting to assess the spreading and penetration of pure water. The pH, conductivity, and wetting rate were measured on 4 samples per treatment.

2.6. Statistical Analysis

Normality of data was confirmed, and two-way ANOVA analysis was carried out using GLM procedure of the SAS 14.1 software (SAS Institute, Campus Drive Cary, NC, USA). The effect of species and temperature on the different charcoal characteristics (density, MC, ash and volatile content, C, H, N, S, O, C/N, C/H, compression strength, conductivity, pH, and wetting rate) were tested. The statistical differences between means were determined by the Tukey test. The analysis of variance and the Tukey tests were performed with the SAS software 14.1 (SAS Institute Inc., Cary, NC, USA). Principal component multivariate analysis (PCA) was performed for the relationship between the parameters evaluated of charcoal and pyrolysis temperatures, and two principal components were established in the cluster grouping.

3. Results and Discussion

3.1. Pyrogenic Carbonaceous Materials (PCMs) Production

The variations in yields of PCMs with temperature are presented in Table 1. The yield decreased with increasing temperature and varied from 45–55% at 300 °C to 30–35% at 500 °C. The lowest PCM percentage presented in *D. panamensis* and the other three species (*G. arborea*, *H. alchorneoides*, and *T. grandis*) presented similar values at different temperatures (Table 1). The decrease in yield with temperature can be attributed to greater production of vapors and volatile content [48,49] caused by the elimination of hydrogen and oxygen by dehydrogenation and deoxygenation processes to produce CO₂, CO, H₂, and water [50]. This behavior is consistent with other tropical wood reported by Lima et al. [51].

Table 1. Average of yield percentages of PCMs obtained for four wood residues tested in slow pyrolysis.

Species	Yield Percentages of PCMs				
	300 °C	350 °C	400 °C	450 °C	500 °C
<i>D. panamensis</i>	44.48	33.80	31.42	28.47	28.18
<i>G. arborea</i>	56.63	41.63	37.18	32.97	30.46
<i>H. alchorneoides</i>	50.21	48.11	35.64	32.57	33.69
<i>T. grandis</i>	46.74	51.00	36.56	33.78	32.47

3.2. Physical and Mechanical Properties of PCM

Values of density, MC, and compression strength were higher in parent wood than charcoal produced at different temperatures (Figure 1). In charcoal of different temperatures,

no differences were found in density and compression strength, except for the density of *T. grandis* at 400 °C, 450 °C, and 500 °C and the MC of *H. alchorneoides* at 500 °C, which presented the lowest values (Figure 1a, 1c). It was observed that the MC of the charcoal produced at 400 °C and 500 °C presented higher values than the other three temperatures in all species (Figure 1b).

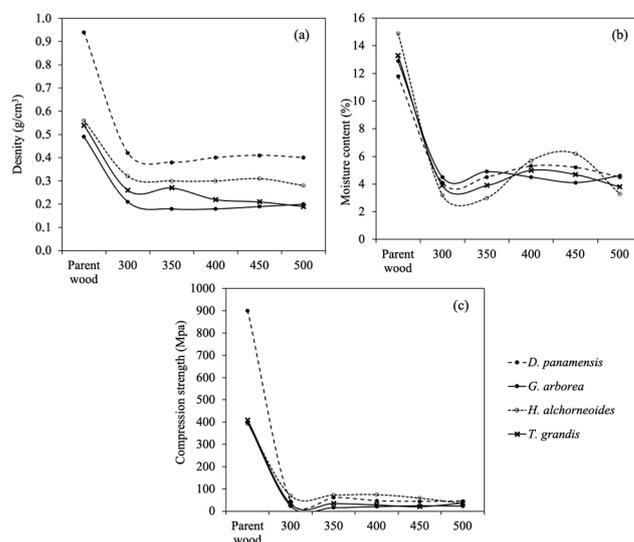


Figure 1. Variation of density (a), moisture content (b), and compression strength (c) for four woody biomass samples tested in slow pyrolysis. Legend: Different letters between different temperatures for same species are statistically different at 99%.

All evaluated parameters of the PCMs, and related to charcoal or biochar, were affected by species, temperature of pyrolysis, and interaction of the two factors, except initial contact angle for biochar properties (Table 1). This result shows that PCMs of the four species studied are different; however, for objective of this study, the results and discussion are focused on the relation to the temperature of pyrolysis.

According to Table 2, charcoal density pyrolyzes at low temperatures (300–350 °C) resulting, in general, in higher density values than charcoal of temperature of 400–500 °C. Moya et al. [24] attributed the higher density at 300–650 °C to incomplete pyrolysis of parent wood, leading to non-charring. Wang et al. [7] explained that parent wood using low temperatures did not completely pyrolyze the cellulose and lignin, because the decomposition of these polymers occurred between 450 and 500 °C.

On the other hand, there is positive high correlation between parent wood density and charcoal density [49]. *D. panamensis* parent wood, with the highest density, produced charcoal with a higher density, in contrast to *G. arborea* parent wood, characterized by the lowest density, which produced charcoal with a lower density (Table 2).

On the other hand, compression strength is related to density [19,49]. As expected, the species with a high density presented higher compression values.

MC ranged from 3 to 6% (Table 1), despite the limited affinity of charcoal with water [52]. This result is due to the presence of some functional groups, especially oxygen-containing groups, which present an affinity with water [52,53]. Another important observation was that the MC was different between different species and with temperature (Table 1). These results confirm that each species generally has its own pyrolysis condition with temperature, resulting in different effects on functional groups [7].

Table 2. F-values for ANOVA of different charcoal characteristics for four wood residues tested in slow pyrolysis.

Parameter	Species Effect	Site Effect	Interaction
Density	443.02 **	444.80 **	16.02 **
Moisture content	3.06 *	1821.77 **	28.83 **
Compression strength	98.51 **	1438.63 **	93.68 **
GCV	5.26 **	113.24 **	1.81 **
Volatile matter (%)	130.20 **	776.24 *	4.91 **
Ash (%)	78.35 **	18.56 **	8.25 **
C (%)	16.13 **	493.50 **	3.16 **
H (%)	5.02 **	399.83 **	3.32 **
O (%)	33.21 **	459.26 **	3.34 **
N (%)	14.59 **	35.28 **	9.30 **
C/N ratio	16.65 **	30.04 **	10.19 **
C/H ratio	2.51 NS	239.14 **	3.72 **
O/C molar	20.36 **	982.02 **	2.49 **
H/C molar	5.07 **	1273.33 **	3.39 **
Conductivity ($\mu\text{S}/\text{cm}$)	104.83 **	3.07 *	4.69 **
pH	133.34 **	104.37 **	14.42 **
Initial contact angle	1.11 NS	1.60 NS	5.58 **
Wetting rate	5.55 **	7.88 **	4.22 **

Legend: ** represents statistically significant at 99% of confidence, * represents statistically significant at 95% of confidence and NS presents not statistically significant.

3.3. FTIR Analysis

The FTIR absorption spectrum was used to note changes in chemical compositions of the charcoals fabricated at different pyrolysis temperature, and the respective parent woods (Figure 2). *D. panamensis* charcoal pyrolyzed at 300 °C presented a similar FTIR spectrum and peak intensity to the respective parent wood (Figure 2a). In other species and at other temperatures, the intensity of peak decreased due to the presence of an O-H bond or hydroxyl groups in the bands of 3400 cm^{-1} and 2910 cm^{-1} , and the peak intensity at 1100 cm^{-1} evidenced the greater change in the O-H bond (Figure 2b,c).

Another important chemical modification was found in charcoal produced at higher temperatures. The FTIR spectrum shows some differences in the peak between parent wood and charcoal pyrolyzed at 350 °C. A decrease in peak intensity at 2910, 2160, 1700, 1630, 1600, 1434, 1375, 1206, 1111, 1032, 900, and 810 cm^{-1} was observed (Figure 2c). However, for charcoal produced at 350 °C, 400 °C, and 450 °C, a decrease in peaks at 2910, 2160, 1630, and 1600 cm^{-1} was observed and the peak intensities at 1700, 1434, 1375, 1206, 1111, 1032, 900, and 810 cm^{-1} were not presented (Figure 2c). Finally, for charcoal pyrolyzed at 500 °C, the peak intensities at 2910, 2160, and 1600 cm^{-1} were not presented, as they were presented in charcoal pyrolyzed at 350–450 °C (Figure 2c).

According to Section 2.3.4, the signal detected at 1700 and 1600 cm^{-1} is associated with C=O of hemicellulose and cellulose degradation and conjugation of the C=O bond with the aromatic ring (1600–1630 cm^{-1}) in all species and temperatures is higher than at 350 °C. The C=O peak occurs due to wood dehydrogenation in lignin and hemicellulose during the pyrolysis process [39]. However, the intensity of the C=O peak (1700 cm^{-1}) was low in temperatures of 450 °C and 500 °C in all species (Figure 2), so these results confirm higher wood dehydration in charcoal [39].

The decreasing CH_2 (1434 cm^{-1}), which corresponds to the aromatic nuclei in lignin, indicates that the lignin of the parent wood is scarcely degraded during pyrolysis [39] in all species, indicating that there is a lignin degradation. The intensity was higher at 300–350 °C than higher temperatures, and the lignin was not completely degraded during pyrolysis at the temperatures of 300–350 °C.

The C-O (stretching) peak (1206, 1032, and 1111 cm^{-1}), which is linked in bonds in acids phenols and ester groups in the precursor fiber [40], was maintained in different

charcoals. These C-O groups are difficult to identify in carbon components because they are ascribed to acids, alcohols, phenols, ethers, and ester groups [40].

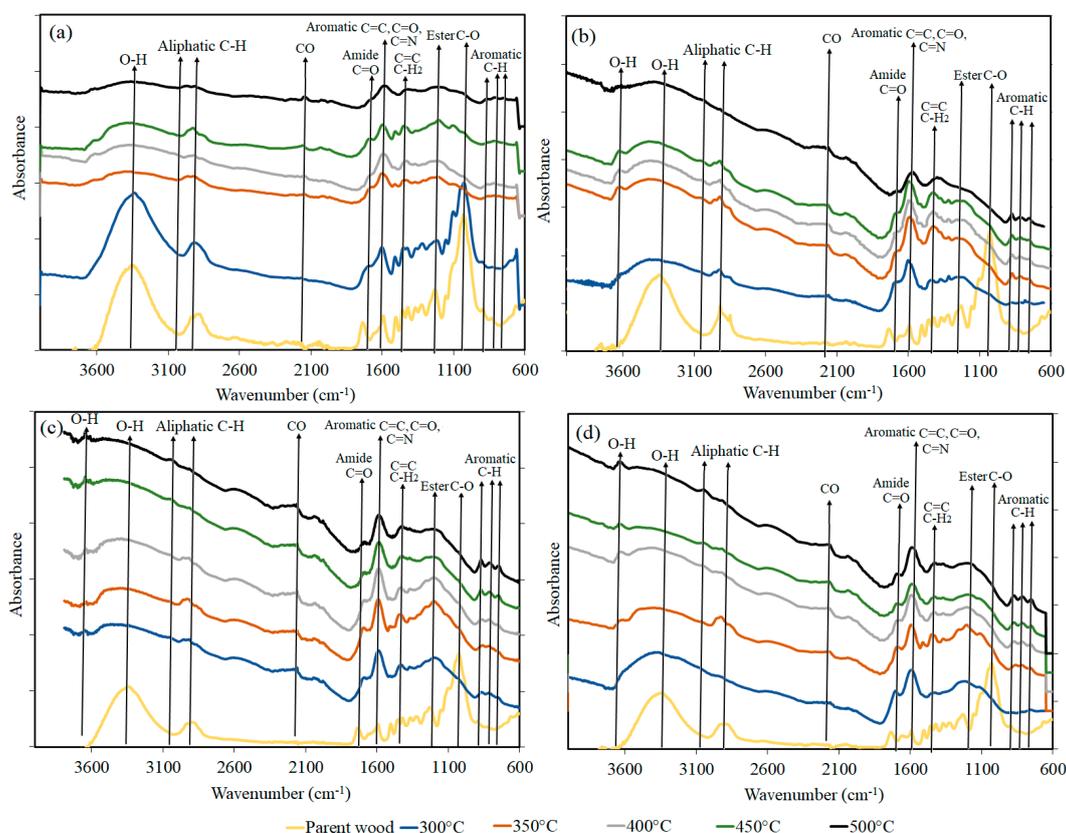


Figure 2. FTIR spectra of (a) *D. panamensis*, (b) *G. arborea*, (c) *H. alchorneoides*, and (d) *T. grandis* charcoals obtained at different pyrolysis temperatures.

Although intensity increased in the bands below 780 and 810 cm^{-1} in all species and temperatures (Figure 2), according to Section 2.3.4, there are greater changes in cellulose and lignin. The presence of these bands is attributed to complete destruction of the pyranoid rings in cellulose (band 897 cm^{-1}) [39] and complete collapse of the net structure of lignin [54], which originates in the skeletal vibration, as well as the change in the relative intensity of trio-bands at these intensities.

In all four cases, the O-H simple bond peak near 3400 cm^{-1} diminished with increasing pyrolysis temperatures, revealing a loss of the hydroxyl function during pyrolysis, which would, in turn, imply dehydration with ensuing loss of aliphatic -H. A similar trend occurred regarding the aliphatic C-H at 2900 cm^{-1} , signaling the loss of simple C-C bonds. Concomitantly, the vibrations at 1400 cm^{-1} and 1600 cm^{-1} related to C=C double bonds increasing in importance with pyrolysis temperatures. These trends confirm a gain in aromaticity and concomitant loss of other functions, with increasing pyrolysis temperatures, as discussed by Sahoo et al. [17].

In summary, according to below results, FTIR spectrum analysis shows important changes in the structure linked to the groups OH, CH₂, C=O, C-O-C, and other less important groups such as CH₃. Basically, at low temperatures ($300\text{--}350\text{ }^{\circ}\text{C}$), the changes associated with lignin and cellulose still present in groups associated with the oxygen (OH, C=O, and C-O-C) of cellulose and aromatic rings of lignin (CH₂), which demonstrates the lack of pyrolysis of the parent wood. Nevertheless, the high temperatures and the pyrolysis completely transformed the parent woods, as revealed by the peak intensity decreases.

3.4. Energy and Chemical Properties

The parent woods presented the statistically highest values of volatile matter, H, O, and C/N and the statistically lowest values of GCV, ash content, C, N, and C/H, (Table 3). For GCV, the highest values were observed in the temperatures of 400 °C, 450 °C, and 500 °C, and no differences were observed between these temperatures (Table 3), and the charcoal from temperatures of 300 °C and 350 °C presented similar values of GCV (Table 3). The volatile matter and H and O of charcoal from temperatures of 300–350 °C have the highest values in most species, while the charcoal at 450 °C and 500 °C show the statistically lowest values. Ash and C content at 400 °C, 450 °C, and 500 °C presented the highest values, while the lowest values were presented at 300 °C and 350 °C in general for all four species (Table 3). No statistical differences were observed in N content of charcoal of *G. arborea*, while charcoal produced from *D. panamensis* and *T. grandis* presented the highest of N content at 300 °C. N content in *H. alchorneoides* was different at all temperatures tested (Table 3). The C/N ratio for *D. panamensis* and *G. arborea* was statistically similar at all temperatures, but charcoal produced from *H. alchorneoides* and *T. grandis* at different temperatures showed some differences at 300 °C and 350 °C (Table 3). Finally, C/H ratio presented the lowest values at temperatures of 300 °C and 350 °C and the highest values were found in temperatures of 400–500 °C in all species (Table 3).

Energy properties evaluated reflected the variety in the species tested and variety in target temperature (Table 3). When GCV values in charcoal exceed 28–30 MJ/kg, those are considered appropriate pyrolysis conditions [52]; therefore, the temperatures of 450 °C and 500 °C can be interpreted as the optimum temperature levels. On the contrary, the temperatures of 300–350 °C produce charcoal with GCV lower than 28–30 MJ/kg, showing that these temperatures are not adequate for charcoal production. The determination of volatile matter confirms that the low pyrolysis temperature (300–350 °C) is not recommended, as this parameter presents the highest values in most species, while the charcoal produced at 450 °C and 500 °C shows the statistically lowest values.

Ash is formed by inorganic matter and in small percentages by organic matter, and the variation in the structural components and inorganic constituents is due to the pyrolysis temperature and intrinsic chemical composition of a species [7,55]. According to Table 3, a higher ash content is found at the higher temperatures. A high percentage of ash is not desirable for energy purposes, but it can be beneficial if the ash is used as biochar, due to the high content and variety of macro- and micro-elements [55]; therefore, ash from high pyrolysis temperatures can be convenient for its higher ash content.

The variation in chemical composition shows the effect of temperature of pyrolysis. As expected, carbon content increased with temperature, but O and H content decreased; therefore, C/H increased with increasing pyrolysis temperatures and O/C molar and H/C molar decreased with temperature (Table 3). Sahoo et al. [17] mentioned that these chemical changes with temperature occurred by improvement in the carbonization and aromatization processes, meaning that the O/C and H/C ratios decreased, as presented in this study (Table 3). In addition, the chemical changes of C, H, and O produced changes in O/C molar and H/C molar ratios [55], as was observed in the present study (Table 3).

The van Krevelen diagram presents the importance of H and O ratio on GVC (Figure 3). The parent wood is similar to different types of biomasses, but during pyrolysis, the wood produces physicochemical changes in C, H, N, and O content (Table 3), which can be observed in the different feedstocks of energy present in the van Krevelen diagram. First, the pyrolysis temperature increase produces a decrease in H/C and O/C ratios, then the charcoal is closer to lignite and coal according to the van Krevelen diagram. Second, for the lowest temperature (300–350 °C), although charcoal of these temperatures is closer to coal, it is also closer to lignite, but charcoal produced at 450–500 °C appears closer to anthracite (Figure 3). This diagram confirms that higher temperatures are better for energy purposes.

Table 3. Energy characteristics of different charcoal characteristics for four wood residues tested in slow pyrolysis.

Species	Variable	Pyrolysis Temperature (°C)					
		Parent Wood	300	350	400	450	500
<i>D. panamensis</i>	GCV (MJ/kg)	19.1 ^C	29.1 ^B	29.1 ^B	30.1 ^A	30.9 ^A	29.9 ^A
	Volatile matter (%)	87.9 ^D	32.2 ^A	31.8 ^A	28.0 ^B	24.4 ^C	28.5 ^B
	Ash (%)	1.5 ^B	1.3 ^B	1.5 ^B	2.2 ^A	2.0 ^A	2.0 ^A
	C (%)	47.3 ^C	76.1 ^B	77.6 ^B	80.2 ^A	82.0 ^A	81.98 ^A
	H (%)	6.6 ^C	4.1 ^A	4.1 ^A	3.7 ^B	3.5 ^B	3.7 ^B
	O (%)	44.5 ^C	17.9 ^A	16.4 ^A	13.6 ^B	12.2 ^B	12.03 ^B
	N (%)	0.07 ^C	0.6 ^A	0.33 ^B	0.28 ^B	0.30 ^B	0.28 ^B
	C/N ratio	708.0 ^C	142.7 ^A	236.9 ^A	291.8 ^A	279.7 ^A	297.2 ^A
	C/H ratio	7.2 ^D	18.4 ^C	19.1 ^C	21.8 ^B	23.8 ^A	22.32 ^B
	O/C molar	0.73 ^F	0.19 ^D	0.17 ^C	0.15 ^B	0.13 ^A	0.13 ^A
H/C molar	1.67 ^D	0.65 ^C	0.63 ^C	0.55 ^A	0.50 ^B	0.54 ^A	
<i>G. arborea</i>	GCV (MJ/kg)	18.4 ^C	28.1 ^B	27.5 ^B	28.6 ^A	29.4 ^A	29.8 ^A
	Volatile matter (%)	93.7 ^D	51.5 ^A	44.7 ^B	43.7 ^B	36.6 ^C	35.6 ^C
	Ash (%)	1.2 ^C	2.8 ^B	3.6 ^A	3.8 ^A	4.0 ^A	2.7 ^B
	C (%)	47.6 ^E	72.2 ^D	77.0 ^C	79.4 ^{BC}	81.5 ^{AB}	84.8 ^A
	H (%)	6.9 ^D	4.7 ^A	4.2 ^A	4.0 ^B	3.8 ^B	3.27 ^C
	O (%)	45.5 ^F	22.8 ^A	18.5 ^B	16.4 ^C	14.4 ^D	11.6 ^E
	N (%)	0.12 ^B	0.23 ^A	0.23 ^A	0.23 ^A	0.26 ^A	0.25 ^A
	C/N ratio	412.8 ^B	330.3 ^A	350.1 ^A	359.6 ^A	324.3 ^A	346.3 ^A
	C/H ratio	6.9 ^E	15.3 ^D	18.4 ^C	20.3 ^B	21.6 ^B	26.2 ^A
	O/C molar	0.72 ^F	0.24 ^E	0.18 ^D	0.16 ^C	0.13 ^B	0.10 ^A
H/C molar	1.73 ^F	0.78 ^E	0.65 ^D	0.60 ^C	0.56 ^B	0.46 ^A	
<i>H. alchorneoides</i>	GCV (MJ/kg)	18.6 ^C	28.0 ^A	28.1 ^A	29.6 ^A	29.4 ^A	29.4 ^A
	Volatile matter (%)	83.4 ^D	28.5 ^A	29.4 ^A	25.2 ^B	29.6 ^A	17.9 ^C
	Ash (%)	1.6 ^B	1.3 ^B	1.4 ^B	1.5 ^B	2.0 ^A	1.6 ^B
	C (%)	46.9 ^C	73.2 ^B	73.0 ^B	77.4 ^A	75.4 ^A	78.8 ^A
	H (%)	6.5 ^D	4.4 ^A	4.1 ^A	3.8 ^B	3.5 ^C	3.5 ^C
	O (%)	46.4 ^E	22.2 ^A	22.6 ^A	18.5 ^D	20.8 ^B	17.4 ^D
	N (%)	0.13 ^E	0.24 ^D	0.27 ^C	0.31 ^A	0.29 ^B	0.31 ^A
	C/N ratio	371.7 ^C	306.6 ^A	270.5 ^B	254.6 ^B	266.4 ^B	261.5 ^B
	C/H ratio	7.2 ^D	16.9 ^C	17.8 ^C	20.5 ^B	21.6 ^A	22.8 ^A
	O/C molar	0.74 ^D	0.23 ^C	0.23 ^C	0.21 ^B	0.18 ^A	0.17 ^A
H/C molar	1.67 ^F	0.72 ^E	0.67 ^D	0.59 ^C	0.56 ^B	0.53 ^A	
<i>T. grandis</i>	GCV (MJ/kg)	19.6 ^C	27.6 ^B	27.4 ^B	28.6 ^A	28.2 ^A	30.4 ^A
	Volatile matter (%)	84.0 ^E	29.3 ^B	34.8 ^A	26.7 ^C	23.8 ^D	22.1 ^D
	Ash (%)	1.2 ^B	2.6 ^A	3.1 ^A	2.4 ^A	2.2 ^A	2.6 ^A
	C (%)	47.0 ^D	74.7 ^C	72.9 ^C	76.6 ^B	80.2 ^A	82.1 ^A
	H (%)	6.7 ^C	4.2 ^A	4.4 ^A	3.8 ^B	3.6 ^B	3.5 ^B
	O (%)	46.2 ^F	20.6 ^A	22.4 ^A	19.2 ^B	15.8 ^C	14.0 ^D
	N (%)	0.14 ^D	0.41 ^A	0.24 ^C	0.31 ^B	0.31 ^B	0.30 ^B
	C/N ratio	346.0 ^D	188.9 ^C	310.1 ^B	248.7 ^A	263.2 ^A	274.8 ^A
	C/H ratio	7.0 ^E	18.4 ^{CD}	16.5 ^D	20.2 ^B	22.3 ^{AB}	23.3 ^A
	O/C molar	0.74 ^E	0.21 ^{CD}	0.23 ^D	0.19 ^C	0.15 ^B	0.13 ^A
H/C molar	1.72 ^F	0.67 ^E	0.73 ^D	0.59 ^C	0.54 ^B	0.52 ^A	

Legend: Different letters for five temperatures and parent wood in same species mean statistical difference at 99% level of confidence.

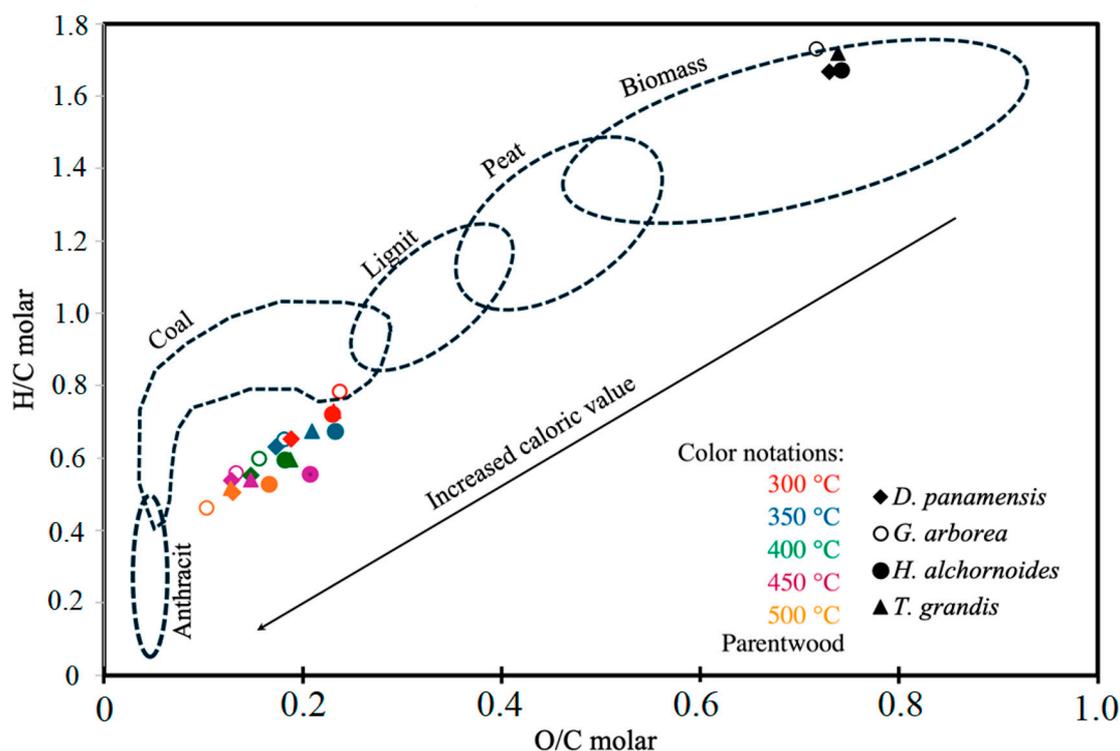


Figure 3. Van Krevelen of different charcoal characteristics for four wood residues tested in slow pyrolysis.

3.5. Thermogravimetric Analysis

The DTG/TG diagram for parent wood and charcoals produced at different pyrolysis temperatures is presented in Figure 4. DTG (Figure 4a,c,e,g) and TG (Figure 4b,d,f,h) for charcoal show the complete transformation of parent wood to charcoal. The maximum decomposition rates occurred between 150 °C to 480 °C, with a shoulder at 325 °C and a light increment after 415 °C; these decomposition temperatures are evidence of the decomposition of cellulose, hemicellulose, and lignin, respectively [56]. When they appear in the charcoal thermograms, they show that these components of the parent wood had not been completely pyrolyzed. For the parent woods, remanent mass was around 20%; in the charcoals, remanent mass was over 60% (Figure 4b,d,f,h). For charcoals, two stages were observed: (i) from 25 °C to 130 °C, where the charcoals dehydrated [57] and other low molecular substances were removed from the surface [56]. The weight loss was approximately 3–6% for the charcoals (Figure 4b,d,f,h), in agreement with values reported in Table 2 for MC; and (ii) from 150 °C and 730 °C, the DTG/TG curves of the charcoals present some differences and trends: the maximum decomposition peak (Tmax) is displaced to higher temperatures with increasing pyrolysis temperatures (Figure 4a,c,e,g). Thus, the charcoal produced presented a Tmax at 517 °C, 575 °C, 620 °C, 620 °C, and 630 °C, for 300 °C, 350 °C, 400 °C, 450 °C, and 500 °C, respectively (Figure 4a).

- (i) Remanent mass increased with increasing pyrolysis temperatures (Figure 4b,d,f,h);
- (ii) Shoulders at 250 °C and 375 °C were observed in charcoal produced from *D. panamensis* (Figure 4a), *G. arborea* (Figure 4c), and *H. alchorneoides* (Figure 4e) obtained at 300 and 350 °C, signaling incomplete carbonization and, thus, evidencing the presence of celluloses, hemicellulose, and lignin in unchanged wood components [56];
- (iii) A small peak appeared near 700 °C in the DTG of *D. panamensis*, *G. arborea*, and *H. alchorneoides*, and was particularly visible in the charcoals obtained at 450 and 500 °C (Figure 4a,c,e).

Charcoal decomposition or maximum devolatilization rate occurs between 200 °C and 750 °C, where functional groups of low thermal stability, such as carboxyl, carbonyl, and aliphatic hydrocarbon groups, are removed [56,58]. The maximum devolatilization

rate is observed between 450 °C and 550 °C for different species, which is evidence of the formation of the aromatic ring and the splitting of more resistant side groups [39]. Tmax varies with pyrolysis temperature and species (Figure 4a,c,e,g), and this parameter increases in *D. panamensis* (Figure 4a) and *H. alchorneoides* (Figure 4e) between 300 °C and 400 °C; meanwhile, the maximum devolatilization rate (peak height) decreases with pyrolysis temperature. The charcoals obtained at 450 °C and 500 °C, in turn, present similar temperature and maximum devolatilization rates for the two species (Figure 4a,e). The DTG curves of *G. arborea* show similar Tmax values for charcoals produced between 300 °C and 450 °C, while the maximum devolatilization rate decreases with pyrolysis temperature (Figure 4c). The Tmax of the 500 °C *G. arborea* charcoal reveals a distinctive devolatilization peak in the vicinity of 650 °C (Figure 4c). Exceptionally, the *T. grandis* charcoals show a Tmax average of 625 °C, and the maximum devolatilization rates decrease with pyrolysis temperature, as in the other cases (Figure 4g).

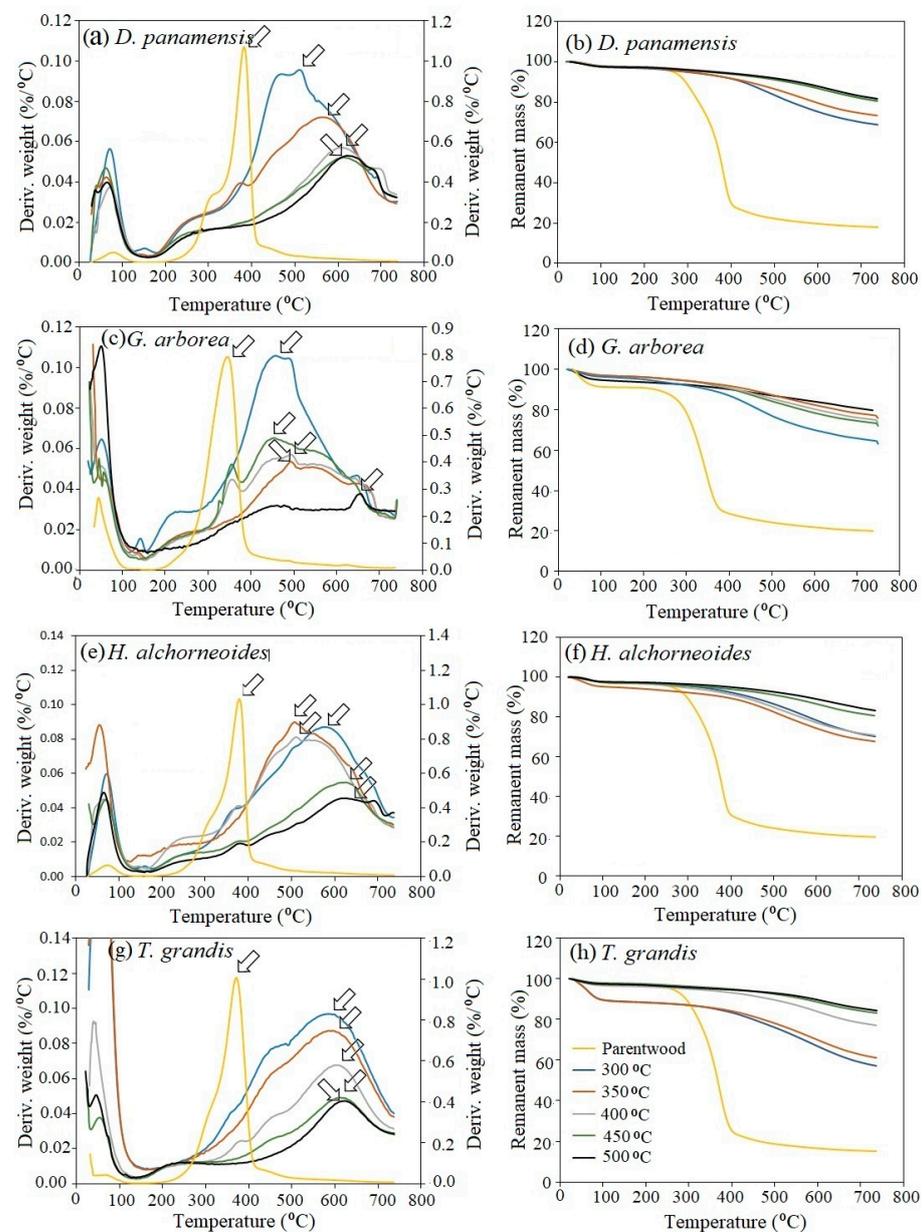


Figure 4. TGA/DTG curves of different charcoal characteristics for four wood residues tested in slow pyrolysis. Legend: Arrows show Tmax.

When comparing among the species, it becomes evident that *T. grandis* charcoals maintained a similar Tmax, while the other charcoals presented Tmax values varying between 475 °C and 630 °C. The increase in Tmax in charcoal was attributed to the fact that this charcoal produced at low temperature was relatively unreactive. Remarkable weight losses did not occur when the temperature reached 340 °C, meaning that char formation began at a higher temperature. The peaks in the devolatilization rates at these temperatures produced fixed carbon in the DTG curves and volatile matter occurred before the maximum devolatilization rate, between 100 °C and 340 °C. Thus, considering this aspect, for *D. panamensis* (Figure 4a), *H. alchorneoides* (Figure 4e), and *T. grandis* (Figure 4g), combustion of volatile matter that remains in the coal occurs mainly at temperatures from 300 °C and 400 °C, but combustion of fixed carbon occurs at temperatures between 450 °C and 550 °C.

3.6. Biochar Properties

In the evaluation of charcoal as biochar, higher conductivity was found in *G. arborea* and *D. panamensis*, but charcoal of *H. alchorneoides* and *T. grandis* presented the lowest values (Table 4). However, some differences can be found according to different temperatures of pyrolysis: (i) the conductivity was higher in charcoal of *G. arborea* produced between 300 °C and 400 °C, followed by *D. panamensis* and *H. alchorneoides* charcoal, and the lowest value of conductivity was observed in biochar of *T. grandis*. But when biochar was produced between 450 °C and 500 °C, conductivity values were different between species: the highest values were in charcoal of *D. panamensis*, and the lowest values were found in biochar of *H. alchorneoides* (Table 4).

Table 4. Electrical conductivity and pH of different biochar characteristics for four wood residues tested in slow pyrolysis.

Species	Variable	Pyrolysis Temperature (°C)				
		300	350	400	450	500
<i>D. panamensis</i>	Conductivity (µS/cm)	237.3 ^A	240.3 ^A	337.3 ^A	319.3 ^A	297.3 ^A
	pH	6.5 ^B	6.8 ^B	7.7 ^A	7.8 ^A	7.7 ^A
<i>G. arborea</i>	Conductivity (µS/cm)	286.3 ^A	308.3 ^A	299.0 ^A	163.5 ^B	291.0 ^A
	pH	6.2 ^B	9.53 ^A	9.42 ^A	8.69 ^A	9.03 ^A
<i>H. alchorneoides</i>	Conductivity (µS/cm)	106.8 ^A	104.0 ^A	85.0 ^B	51.0 ^C	61.0 ^C
	pH	5.7 ^{CD}	5.2 ^D	6.6 ^{BC}	7.6 ^{AB}	7.6 ^A
<i>T. grandis</i>	Conductivity (µS/cm)	107.0 ^A	91.3 ^B	71.3 ^D	73.3 ^D	71.3 ^D
	pH	5.3 ^D	5.7 ^D	6.4 ^{BC}	7.0 ^{AB}	7.6 ^A

Legend: Different letters for five temperatures and parent wood in same species mean statistical difference at 99% level of confidence.

Conductivity is a measure of the amount of salts in biochar solution and based on the principle that a solution with a higher concentration of salts has a greater ability to conduct an electrical current [59].

Therefore, when charcoal is applied in soil, for a biochar propose, with high rates of conductivity, this may adversely affect salt-sensitive plants [60] and can reduce seed germination and crop yields [61]. Therefore, biochar produced with *D. panamensis* and *G. arborea* must be managed or applied in different forms or proportions in soil than the biochar of *H. alchorneoides* and *T. grandis*.

The conductivity varied with temperatures for all species. No differences were observed among temperature in the charcoal of *D. panamensis*; the lowest conductivity was presented in biochar of *G. arborea* at a temperature of 450 °C and no differences were found in other temperatures (Table 4). *H. alchorneoides* and *T. grandis* biochar presented the highest conductivity values for temperatures of 300–350 °C, and the lowest values were observed in biochar at 450 °C and 500 °C (Table 4).

The variation in conductivity depending on species and temperature agree with the reviews presented by Singh et al. [59] and Paz-Ferreiro et al. [61], which report that this parameter is related to feedstock and temperature. However, the tendency found in our results disagrees with Singh et al. [59]. They mention that higher temperatures generally have higher conductivity values, however, our results show that the lowest values are found in the highest temperatures, or no differences were found in some species such as *D. panamensis*. Biochar conductivity is related to ash content and pH [59], chemical characteristics of parent wood [62], macro- or micro-nutrients such as potassium [61], and other factors. According to these results, the biochar from each species at each temperature must be applied in relation to its conductivity. The parent wood used presented wide variations in chemical properties [36], which can affect the biochar conductivity produced at different temperatures and other chemical properties, such as macro- or micro-nutrients, which were not measured in the present study.

In relation to pH values, biochar of *G. arborea* presented the highest values of pH, except for at the temperature of 300 °C. The biochar of *D. panamensis* presented pH values lower than *G. arborea* biochar and higher than *H. alchorneoides* and *T. grandis* biochar, which presented the lowest values observed (Table 4). The pH of biochar will positively influence soil pH [63], though its liming effect on soil will vanish over time as soil acidification is a spontaneous process, especially in the tropics. Although biochar from the other three species (*D. panamensis*, *T. grandis*, and *H. alchorneoides*) can be applied according to pyrolysis temperature, biochar from lower pyrolysis temperatures (300–350 °C) could increase soil acidity due to having a lower potential liming effect and to their liability to progressively decompose in the soil, increasing soil acidity. Meanwhile, biochar produced at the higher temperatures (400 °C to 500 °C) cannot affect adversely soil acidity, since the pH values are close to the ideal pH range for soils, which is from 6.0 to 6.5.

The variation in pH values with temperature in the different species studied (Table 4) is attributed to different decomposition processes of biomass with temperature [64]. Specifically, it was observed that pH values of different species sampled increased with temperature (Table 4). Ding et al. [65] mentioned that pH values were positively correlated with temperature from 300 °C to 600 °C; thereafter, pH became constant [66]. In general, it was established that pH was associated with inorganic alkalis content and the formation of carbonates, and these components increased with pyrolysis temperature [65]; in addition, higher temperatures favor functional groups and ash content [67]. For the polymer decomposition of wood with increasing temperature, acidic functional groups (-COOH) and appearance of basic functional groups are removed during pyrolysis [68] and the increasing temperature occurs due to the separation of the organic matrix, mainly alkali salts [65].

Initial contact angle is a parameter used to predict the biochar hydrophobicity; higher values of this angle means that biochar is hydrophobic [69], meaning that its surface energy is lower, therefore, the biochar has less attraction to water at the beginning of water exposure. The results found in the study show that low pyrolysis temperatures (300–350 °C) produced the highest initial contact angle (114–117°) for *T. grandis* and *H. alchorneoides* biochar. Meanwhile, biochar of *D. panamensis* presented the lowest values (112–113°) and biochar of *G. arborea* presented intermediate values (112–114°, Figure 5a). On the contrary, for biochar produced at 400 °C and 500 °C, it was found that charcoal of *T. grandis* presented the highest values (119–121°), followed by the biochar of *H. alchorneoides* and *D. panamensis* (117–119°) and the biochar of *G. arborea* presented the lowest values of initial contact angle (114–116°).

The relationship between water and biochar can be understood using different properties, and wettability is one of them [69]. Water rate was the highest in *D. panamensis* biochar at the five pyrolysis temperatures evaluated, while the other three species presented similar values at all pyrolysis temperatures, especially above 400 °C (Figure 5b). The variation in wetting rate was different in each species: this value increased with temperature in *D. panamensis* and *H. alchorneoides* biochar, but the variation was different in *T. grandis* and *G. arborea* biochar, with increasing wetting rate values from 300 °C to 350 °C and from

450 °C and 500 °C; no variation was observed between 350 °C and 450 °C (Figure 5b). Thus, according to these results, the biochar of *D. panamensis* presented lower hydrophobicity than those of other species across time, meaning this biochar could take up water faster than those of *H. alchorneoides*, *G. arborea*, and *T. grandis*, though they showed similar water absorption rates.

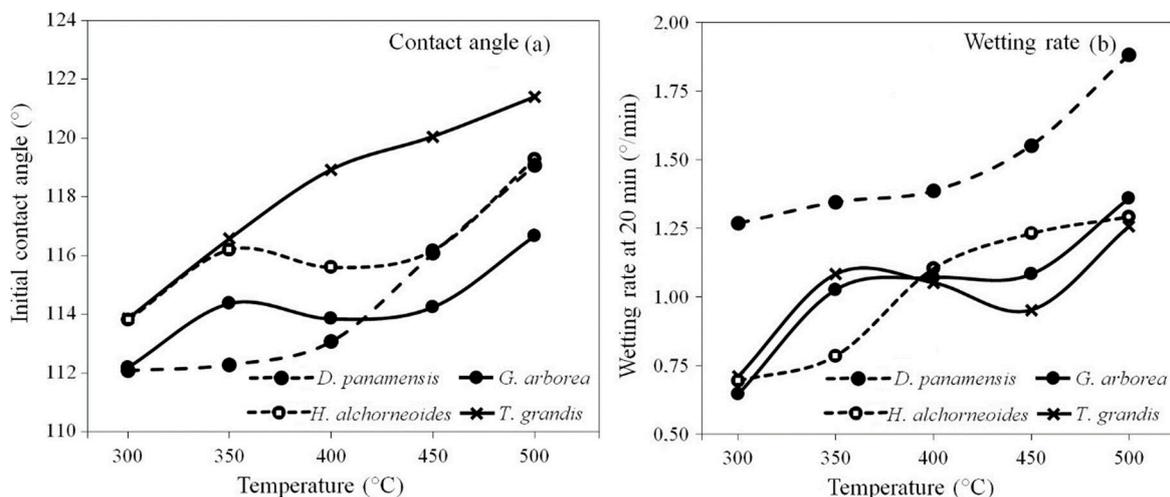


Figure 5. Initial contact angle (a) and wetting rate at 20 min (b) of different biochar characteristics for four wood residues tested in slow pyrolysis.

Figure 5 shows similar trends for all wood species; generally, higher hydrophobicity and wetting rate is observed with higher temperature. According to Rasa et al. [70], the relationship between biochar and water is a very complex phenomenon, and when the presence of aliphatic functional groups (C-H) is greater, the biochar is more hydrophobic; however, they also mention that biochar produced at temperatures lower than 500 °C present lower surface area values, lower presence of aliphatic functional groups, and increased porosity, thereby enhancing wettability of biochar [71], measured in this study by wetting rate. Besides this relationship between biochar and water, each species presents a different decomposition process of biomass with temperature during pyrolysis [68], which affects the aliphatic functional groups, with a lower presence of aliphatic groups at higher pyrolysis temperatures. This would lead to a variation in the initial contact angle or hydrophobicity and wetting rate of each species studied. Sahoo et al. [17] mentioned that increasing temperature produces an enhancement in carbonization, the aromatization process, and a decremental nature of O/C and (N + O)/ration ratios; then, biochar becomes more hydrophobic and non-polar, and there is a hydrophobicity that increases with temperature, as observed in the species studied (Figure 5a). Nevertheless, increased porosity increased wetting rate [69], as observed in the present study (Figure 5b).

3.7. Multivariate Analysis

Multivariate principal components analysis shows that the first two components explain the variability in 62.0%: 39.0% and 26.0% for principal component 1 (CP-1) and 2 (CP-2), respectively (Table 5). CP-1 was related to GCV, C, H, and O content, C/H ratio, and pH and wetting rate (Table 5). CP-2 was only related to charcoal density for physical properties, volatility and ash content for charcoal characteristics, and C/N for chemical characteristics (Table 5). Scatterplots of principal component 1 and principal component 2 show that three different groups formed (Figure 6):

- The first group groups charcoal of *H. alchorneoides* and *T. grandis* pyrolyzed at 300 °C and 350 °C (low temperatures) and that of *D. panamensis* of pyrolysis temperature 300 °C. This group is associated with pH and C/N ratio;

- The second group is formed by all the charcoals of *G. arborea*, and is associated with O and H content, C/H ratio, MC, and GCV;
- The third group includes the charcoals of *H. alchorneoides*, *T. grandis*, and *D. panamensis* pyrolyzed at 350 °C to 500 °C; it is associated with contact angle, wetting rate, ash, N and C content, compression force, conductivity, volatility, and density.

Table 5. Statistical parameters of CP-1 and CP-2 and their correlations with different characteristics measured in charcoal of four tropical wood from plantations.

Parameters	Variable	CP-1	CP-2
Statistical parameters of principal components	Eigenvalue	6.06	4.47
	% Total of variance	36.0	26.0
	Total cumulative	36.0	62.0
Correlations of characteristics measured and principal components			
Variables	Parameters	CP1	CP2
Physical and mechanical properties	Density	−0.01	−0.77 *
	Moisture content	0.37	0.01
	Compression strength	−0.27	−0.65
Charcoal characteristics	GCV	0.80 **	−0.38
	Volatility	−0.31	0.77 *
	Ash (%)	0.18	0.86 **
Chemical characteristics	N	−0.15	−0.68
	C	0.96 **	0.02
	H	0.89 **	0.27
	S	−0.09	−0.47
	O (%)	0.95 **	−0.03
	C/N ratio	0.26	0.83 **
	C/H ratio	0.95 **	−0.15
Biochar characteristics	pH	0.76 *	0.50
	Conductivity	0.31	0.37
	Initial contact angle	0.47	−0.12
	Wetting rate	0.77 *	−0.34

Legend: ** Denotes significance of p -value < 0.01 and * denotes significance of p -value < 0.05.

The analysis by principal components confirms important observations that were annotated individually in the variables analyzed previously. In general, it was observed that the lowest temperatures (300 °C and 350 °C) of *T. grandis* and *H. alchorneoides* and the pyrolysis temperature of 300 °C for *D. panamensis* grouped together, indicating that, probably, these conditions are not adequate for pyrolysis, considering that these temperatures present higher values of density, moisture content, compression strength, volatile matter, and H and O content, and lower higher values of C and ash content, conductivity, initial contact angle, and wetting rate and lower pH. In addition, FTIR and TGA show that celluloses and lignin were not completely decomposed at temperature of 300 °C to 350 °C. A second important observation is that the five different charcoals of *G. arborea* were grouped in one cluster, different from the cluster formed by *D. panamensis*, *H. alchorneoides*, and *T. grandis* (Figure 6), and this variation is associated with differences in O and H content, C/H ratio, MC, and GCV. Finally, charcoals produced from *D. panamensis*, *T. grandis*, and *H. alchorneoides* at 400–500 °C were grouped into one cluster, probably indicating that as the best temperature range for pyrolysis of these species.

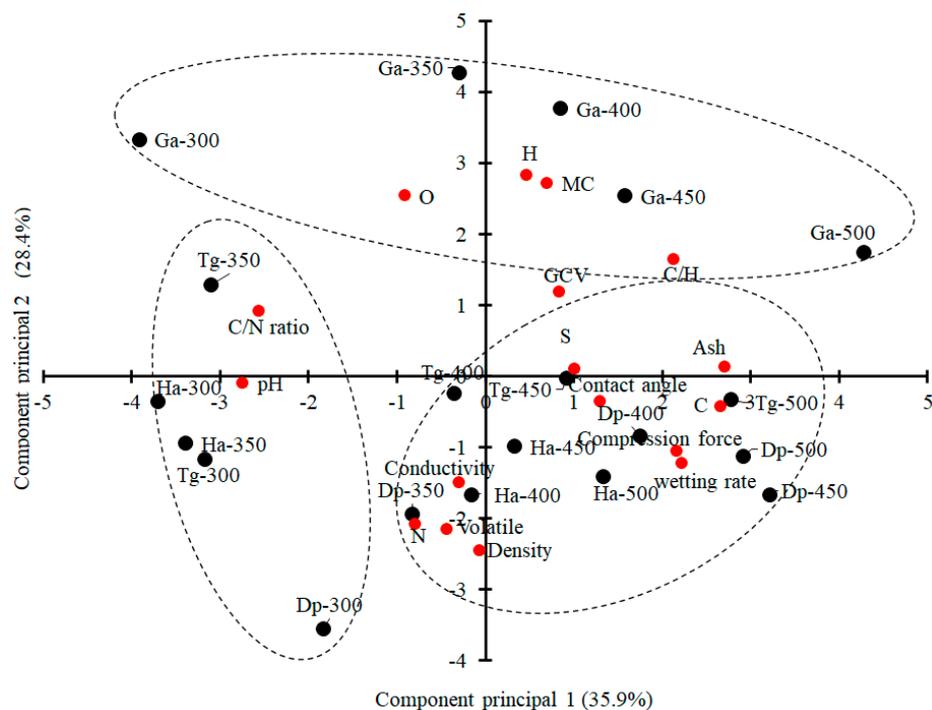


Figure 6. Relationships between principal component 1 and 2 for different biochar characteristics for four wood residues tested in slow pyrolysis. Legend: names of variables are detailed in Table 5.

4. Conclusions

According to the results obtained in the evaluation of properties of pyrogenic carbonaceous materials (PCMs) from four different tropical wood species obtained at five different temperatures, each species*temperature combination had its different effects according to PCM propose. Firstly, each species produced PCM characteristics related to intrinsic proprieties of biomass, and, second, temperatures of 300 °C and 350 °C are not recommended due to the presence of uncharred areas, as demonstrated by FTIR and TGA analysis; besides that, these charcoals present higher values of density, moisture content, compression strength, volatile matter, and H and O content, and lower values of C and ash content, conductivity, pH, initial contact angle, and wetting rate. The charcoals produced between 400–500 °C had contrasting characteristics compared to 300 °C and 350 °C across all species, so that the higher temperatures (400–500 °C) presented better conditions in slow pyrolysis of the species studied. Multivariate analysis confirms that charcoals obtained at the lowest temperatures (300–350 °C) from *D. panamensis*, *Hieronyma alchorneoides*, and *T. grandis* were separated from those obtained at other temperatures, indicating that, probably, (i) these conditions were not adequate for pyrolysis, (ii) the five different pyrolysis attempts of *G. arborea* were grouped in a cluster apart from the other species, with different charcoal/biochar properties, and (iii) the charcoal/biochar produced from *D. panamensis*, *H. alchorneoides*, and *T. grandis* at 400–500 °C were grouped into one cluster, probably indicating that to be the best temperature range for pyrolysis of those species.

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