

Physical-Chemical and Energy Characterization of Residual Biomass from Baru Fruit (*Dipteryx Alata* Vogel)

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Abstract

The use of the fruit of the species *Dipteryx alata* Vogel (baru) is a highly profitable and expanding activity in the Brazilian market, although the almond of the fruit has high commercial value, the processing of the fruit generates about 87% of residual biomass. The objective of this work was to characterize the biomass and biochar obtained from the residual biomass of the fruit. Biomass was characterized in relation to physical, chemical and energetic properties. Subsequently, biomass carbonization was carried out at different rates of final temperatures (450; 550 and 650 ° C). For carbonization, the gravimetric yield in biochar, bio-oil and non-condensable gases was analyzed. Biochar was characterized in relation to chemical, physical and energetic parameters. The apparent density value found for biomass was 0.917 g.cm⁻³ while the biochar varied from 0.723 to 0.768 g.cm⁻³. Regarding the chemical composition of the biomass, the extract value was 9.88%. The total lignin content was 28.29%. For the immediate chemical analysis the values were 0.40 for ash, 18.47% for fixed carbon and 81.34% for volatile materials. The gravimetric yield of biochar at different temperatures ranged from 34.99 to 39.48%, while the yield on condensable gases ranged from 35.36 to 40.33% and the yield on non-condensable gases was around 23.54 to 25, 29%. The calorific value of biomass was 21,176 kJ.kg⁻¹ and biochar up to 35,328a kJ.kg⁻¹. The values found indicate that the residual biomass and biochar from the fruit's pericarp, showed high energy potent.

1. INTRODUCTION

Worldwide, there is a constant search for forms of renewable energy. Faced with this demand, several proposals for the use of new and renewable energy sources arise. Forest biomass due to its high potential for use has aroused interest for energy purposes (Vilas Boas et al. 2010, Pang 2016, Deboni et al. 2019). Biomass is a source of 14% of the energy used in the world (35% of energy in developing countries). In Brazil, according to the National Energy Balance (2022), biomass represents 8.8% of energy use in the country, against 60.2% of the source of hydraulic electricity. The development of traditional conversion processes to more efficient systems, leading to modern energy vectors will bring an important change in the utilization profile and in the volumes of biomass for energy (Martinez et al. 2019).

The new bases for rural development and employment, as well as the global limitations to the growth of the concentration of atmospheric CO₂, will be factors for the increase in the use of energy biomass. However, given the specialties of each type of biomass, it is essential to determine the availability and energy characteristics of the waste considered, and to develop appropriate technology for its management, compatible in ecological, economic and social character (Hurskainen and Vainikka 2016).

Residual biomass can be described as any amount left over from a process of production or exploration, transformation or use (Dionizio, 2019). The rational and efficient use of biomass resources, through the use of modern technologies and the use of waste, is a viable alternative for energy production (Vale and Olsen 2013, Veksha et al. 2019).

Among the uses of residual biomass for energy generation, biochar, in addition to being an environmentally correct practice, can add value to activities already developed, contributing to the environmental, social and economic scope of small communities that withdraw part of their livelihood from plant extraction. Jin and Sutherland (2018) report that the efficient conversion of biomass to biochar is the best short-term strategy to reduce greenhouse gas emissions in the electricity sector from non-renewable sources.

The Brazilian Cerrado region is promising, since it has commercial activities linked to sustainable extraction with native fruit species, which result in hundreds of tons of residual biomass annually without proper destination. Among the most commercially benefited species is *Dipteryx alata* Vogel (baru) (ISPN 2010). The species has intermittent fruiting with an average of 150 kg of fruit per tree. Almonds are consumed from the fruits, which can be used in the preparation of various culinary preparations (Pimentel 2008, Vera and Souza 2009, Rodrigues and Ribeiro 2018). In the beneficiation, the almond is separated from the pericarp, for each 1 ton of processed fruit, 870 kg is considered residual biomass of the process (Viana 2015).

Investigating the potential of new biomass as feedstock for biochar production, its pyrolysis characteristics and energy potential is of relevance mainly for the bioeconomy (Diniz et al. 2021). For this purpose, we focus on the impact of different pyrolysis temperatures on the yield and physicochemical properties of baru endocarp biochar. This research helps to select the optimal pyrolysis temperature to produce specific biochar with desirable characteristics for future applications. The objective of this work was to evaluate the energy potential of this residual biomass from the fruit of the species *Dipteryx alata* Vogel.

2. MATERIAL AND METHODS

The study material used was the pericarp of the fruit of the species *Dipteryx alata* Vogel. The material was collected in an extractive production unit called São Miguel, in the municipality of Anastácio, in the State of Mato Grosso do Sul - Brazil. Drying was carried out in the open air until moisture balance, about 11%. After drying the samples were processed in a Willey mill and classified in sieves, the fraction that passed through the 40 sieve and was retained in the 60 mesh sieve was used for chemical analysis.

2.1 Chemical characterization

The chemical composition was determined from the extractive contents, according to the TAPPI 264 om-88 standard, insoluble lignin, according to the Klason method, modified according to the procedure proposed by Gomide and Demuner (1986), derived from the TAPPI T standard 224 om-88 and soluble lignin, determined by spectrometry, according to Goldschimid (1971). The total lignin content is equivalent to the sum of the two values. The holocellulose content was obtained by adding the levels of extracts, total lignin and ash decreased by 100.

The syringyl/guaiacyl ratio was performed using liquid chromatography after oxidation of the sawdust with nitrobenzene, according to Lin and Dence (1992). The separation of the products from nitrobenzene oxidation was achieved using an LC-18 column. The mobile phase used was acetonitrile / water (1: 6 v / v) with pH equal to 2.6, buffered with trifluoroacetic acid (TFA), detection: UV, 280 nm, T = 40°C, flow: 1.0 mL minute⁻¹, injection 20µl; chromatographic pattern: vanillin for guaiac and syringaldehyde for syringyl.

To determine the elemental analysis (carbon, nitrogen, hydrogen and oxygen) a mass equivalent to 2.5 mg (± 0.5) of absolutely dry sawdust was measured, which was selected in overlapping screens of 200 and 270 mesh, being used the fraction retained in the latter. The equipment used in the analysis was the Vario Micro Cube CHNS-O. Oxygen was quantified by the sum of C, N, H and ash decreased by 100.

Immediate chemical analysis was determined according to NBR 8112 (1983). To determine the content of volatile materials the material was dried at 105 ° C in an oven. 1.0 g of the dry material was weighed in a previously dried and tared porcelain crucible. The crucible was placed in the muffle door at 950°C for 3 minutes, after which the crucible was capped and placed in the muffle at the same temperature for 7 minutes. The calculation was performed according to Eq. 1:

$$TMV = (MI - MA_{950} / MI) \cdot 100$$

Where:

TMV = Content of volatile materials (%);

MI = Initial mass (g);

MA₉₅₀ = Sample mass at 950 °C (g).

The determination of the ash content in a previously dried and tared porcelain crucible, a mass of 1 g of sample was placed and conditioned at a temperature of 750 ° C in a muffle furnace until the material was completely burned. Thus, to determine the percentage of inorganics following Eq. 2:

$$TCZ = (MA_{750} / MI) \cdot 100$$

Where:

TCZ = Ash content (%);

MI = Initial mass (g);

MA₇₅₀ = Sample mass at 750 ° C (g).

The fixed carbon content is an indirect measure and was calculated according to Eq. 3:

$$TCF = (100 - (TMV + TCZ))$$

Where:

TCF = Fixed carbon content (%);

TMV = Content of volatile materials (%);

TCZ = Ash content (%).

2.2 Physical-energetic characterization

The moisture content was calculated with the percentage difference between the initial sample mass and the sample mass after the drying process (NBR 14929, 2003). The apparent density was determined using a scale to measure the displaced volume, for this purpose, the samples were weighed to obtain the wet mass and, later, immersed in mercury to determine the displaced volume (NBR 9165,1985). Thermogravimetric analysis was measured by assessing the gradual rate of mass loss of biomass as a function of the constant increase in temperature. The tests were performed using a TGA analyzer (SHIMADZU DTG60 Series), heating a mass of 4 mg in a nitrogen purge (30 mL min^{-1}), at heating rates of $10^{\circ}\text{C min}^{-1}$ with a final temperature of 1000°C . For the determination of the superior calorific power (PCS) a calorimetric pump was used, approximately 1g was weighed for each sample in a quartz crucible. After determining the sample mass, the crucible was attached to the support of the calorimetric pump. The equipment was turned on and the data regarding the sample weight were entered in a control panel. The calorimetric pump was sealed and coupled to the calorimeter to start the PCS test (NBR 14929, 2003). The determination of the energy density was obtained as a product of the apparent density (g.cm^{-3}) by the superior calorific value (kJ.kg^{-1}).

2.3 Carbonization process

The samples were previously dried at 105°C in a forced air circulation oven. The carbonizations were carried out in an electrically heated muffle using a metal reactor with dimensions of 30 cm in length and 12 in diameter. The biomass was inserted into the container, occupying the maximum of empty spaces. Three final carbonization temperatures were established, 450, 550 and 650°C , and their respective heating rates 1.07; 1.14 and $1.20^{\circ}\text{C/minutes}$ according to Table 1.

Table 1
Carbonization march according to the final carbonization time and temperature.

March ($^{\circ}\text{C/minutes}$)	Temperature ($^{\circ}\text{C}$)								
	150	200	250	300	350	400	450	550	650
1.07	1 hour	1 hour	1 hour	1 hour	1 hour	1 hour	1 hour	-	-
1.14	1 hour	1 hour	1 hour	1 hour	1 hour	1 hour	1 hour	1 hour	-
1.20	1 hour	1 hour	1 hour	1 hour	1 hour	1 hour	1 hour	1 hour	1 hour

The calculations used to determine the yields of the carbonization process are shown in Table 2.

Table 2
Calculations used for yields of the carbonization process.

Equation 1	Equation 2	Equation 3
GYBC = Gravimetric yield on biochar (%)	TBO = Bio-oil content (%)	YNCG = Yield on non-condensable gases (%)
$GYBC = \frac{MB}{MI} \times 100$	$TBO = \frac{MB}{MI} \times 100$	$YNCG = (100 - (GYBC + BO))$
MBC = mass of biochar (g)	MB = Bio-oil mass (%)	GYBC = Gravimetric yield in biochar.
MI = Mass inserted before carbonization (g)	MI = Mass inserted before carbonization (%)	BOC = Bio-oil content (%).

2.4 Data analysis

The experiment was analyzed according to a completely randomized design, with three treatments (final carbonization temperatures), with three repetitions for each carbonization, totaling six sample units. The data were submitted to Bartlett's test to test the homogeneity of variances and Shapiro-Wilk to test normality. Then, the analysis of variance was performed by the F test, with the means being compared by the Tukey test at a significance level of 5%. Statistical analyzes were performed with the aid of the RStudio program (RStudio Team, 2022).

3. RESULTS AND DISCUSSION

3.1 Characterization of biomass

Table 3 shows the values of the chemical characterization of baru biomass. The extract content found was 9.88%, the extracts comprise a series of classes of chemical compounds, among which, classes such as phenolic, are preferable for coal production, due to the carbon composition in its formula, therefore, representing a greater release of energy. These compounds can vary exponentially between species and in different parts of the plant, they are usually concentrated in bark, leaves, flowers, fruits and seeds. The high presence of substances of an aromatic nature, such as extracts and lignin, consequently generates a coal with higher density and higher physical-mechanical quality (Silva et al. 2014).

Table 3
Characterization of baru biomass

Total extracts (%)	9.88
Soluble lignin (%)	2.79
Insoluble lignin (%)	25.50
Syringyl/guaiacyl	0.95
Ashes (%)	0.40
Holocellulose (%)	61.43
Volatiles (%)	81.34
Fixed carbon (%)	18.47
Ashes (%)	0.40
Carbon (%)	53.10
Hydrogen (%)	6.19
Oxygen (%)	39.82
Nitrogen (%)	0.482
Moisture content (%)	11.23
Apparent density (g.cm ⁻³)	0.917
Energy density (kJ.m ⁻³)	19.418
Higher calorific value (kJ.kg ⁻¹)	21.176

The average value of total lignin was 28.29%, this high characteristic can be attributed to physiological issues of fruit formation, since the endocarp houses the seed. Baru biomass showed 0.40% ash. Ashes, as a rule, negatively influence the calorific value of the material, since they are inert structures (Santos 2010, Santos et al. 2011). Low indices of inorganics are qualitative indicatives for charcoal production (Arantes et al. 2013). Santos (2010) reports that the greater the proportion of these constituents, the lower the energy given off by the fuel.

The average value of the holocellulose content was 61.43%, the term holocellulose is used to determine the main carbohydrates in wood, cellulose and hemicelluloses (Santos, 2008), there is an inverse relationship between lignin and holocellulose. Álvarez (2018) reaffirms this idea, where they find 33% lignin and 81% holocellulose for almond bark biomass. Among the value of volatile materials, the fruit showed high concentration, Brand et al. (2013) also reported volatile wood values around 80%. The average value of fixed carbon was 18.47%. The low values are justified by the high proportion of volatile materials present in the material, since it is an inversely proportional relationship.

The apparent density found for baru biomass was 0.917 g.cm^{-3} , the values found are higher than most of the clones currently used in the steel sector for the production of charcoal, such as the *Eucalyptus* clone. *E. urophylla* x *Eucalyptus grandis*, which presents average values of 0.565 g.cm^{-3} in its adult phase (Castro et al. 2016). This is an important parameter for the gravimetric yield in charcoal, directly implying the relation of the space occupied by the reducer inside the blast furnace. Jesus et al. (2017) observed a trend in apparent density as a function of energy density for *Eucalyptus* sp. Energy density is related to the amount of energy present in a given volume. Figure 1 shows the analysis of thermal degradation and its derivative for baru biomass.

In Fig. 1, we can define three distinct phases of thermal degradation of the biomasses of the analyzed fruits. The first stage comprises the initial temperature of 0 to 200 °C, where there is mainly loss of water and some volatiles, with no great distinctions between the biomasses (Chen et al. 2019). In the second phase of 200 to 400 °C we observed that the biomass of baru has a peak of mass loss, more accentuated close to 400 °C, where there is detachment of volatile materials and fixed carbon, representing a greater loss range among the three phases. The last stage comprises the temperature of 400 to 900 °C where it is possible to observe stability of the loss of mass of the baru. According to Talero, Rincón and Gómez (2019) the thermal degradation tends to stabilize since, almost all the organic material was detached, therefore, leaving a high proportion of inorganics, which certainly could explain the thermal resistance at the end of the process.

3.2 Biochar characterization

Table 4 shows that as the carbonization rate increased, there was an increase in humidity, so we can infer that the degree of degradation of the constituents was directly related to the moisture content (Vilas Boas et al. 2010). The density was 0.727 ; 0.723 and 0.768 g.cm^{-3} at the respective temperatures 450; 550 and 650°C. The endocarp of this fruit showed stability in density, as the carbonization rate was intensified.

The values found justify the use of this biomass for charcoal production. Cheng et al. (2016) used charcoal made from sawdust, nutshells and different waste biomasses, using it as an alternative fuel in the sintering process of pig iron. The bottleneck in the use of biomass as an energy source is the unavailability of constant and abundant stock, as well as the absence of technologies that apply to the heterogeneity of waste (Jhan and Soren 2017). Table 3 shows the baru biochar characterization values at different carbonization temperatures.

Table 4
Characterization of baru biochar at their carbonization temperatures.

Biochar characterization	Temperature (°C)		
	450	550	650
Yield (%)	39.48a	36.13b	34.99c
Condensable gases(%)	35.36c	40.33a	39.71b
Non-condensable gases(%)	25.16b	23.54c	25.29a
Volatile materials(%)	23.97a	11.56b	8.19c
Fixed carbon (%)	72.87c	84.34b	87.29a
Ashes (%)	3.16c	4.10b	4.52a
Moisture contente (%)	1.38c	1.54b	2.72a
Apparent density (g.cm ⁻³)	0.727b	0.723b	0.768a
Energy density (kj.m ⁻³)	21,624c	24,739b	27,130a
Higher calorific value (kj.kg ⁻¹)	29,747c	34,222b	35,328a

The average values of gravimetric yield in biochar were high, the final carbonization temperature of 450 °C was significantly higher. The drop in yield is attributed to the loss of mass of the chemical constituents of the fruit with the increase in the final carbonization temperature. The yields found are similar to those obtained from native woods of the caatinga biome (Paes et al. 2012).

Condensable gases were significant at temperatures of 550°C with a value of 40.33%. With the increase in the final carbonization temperature, there was a percentage increase in condensable gases. As the chemical constituents are degraded, several gases are released, part of which is released into non-condensable gases and the other condenses in the form of bio-oil. In the biochar yield, a gradual decrease was observed as the heating rate was high. The temperature was significant for condensable gases. Bio-oil is a by-product of the pyrolysis of lignocellulosic materials, although its economic potential is little explored, it has a wide range of uses (Almeida et al. 2019).

According to the results presented in Table 4, the average ash contents found were significant for the carbonization temperatures of 550 and 650 °C, the lowest value was for the lowest heating rate, 450°C. There was a progressive increase in this content with carbonization, compared to non-carbonized biomass. The values found are close to those obtained by Vilas Boas et al. (2012) for the macaúba fruit. The average values for the volatiles were significant for the different temperatures, there was a decrease as the final temperature increased, and an inverse relationship in the volatile content as for the fixed carbon and ash contents. As for the fixed carbon contents, the average values were significant. It is

known that the fixed carbon content increases with the carbonization temperature, and is positively related to the calorific value of coal.

For the average values of volatile materials, there was a statistical difference, volatile materials tend to decrease, because up to 650 ° C the vast majority of condensable and non-condensable gases have been released. The concentration of fixed carbon was significant for the temperatures tested. The average values obtained were 72.87; 84.34 and 87.29% at temperatures 450, 550 and 650°C respectively.

The biomass of baru obtained the best results in the 550 ° C range, and showed an excellent relationship of gravimetric yield as a function of fixed carbon. Through this relationship, it is possible to verify which is the most suitable final temperature for this material, since it aims at a higher gravimetric yield with a high fixed carbon content.

4. CONCLUSIONS

From the results it can be concluded that the residual biomass and biochar from the species *Dipteryx alata* Vogel presented properties with high energy potential and, therefore, can be used for products aimed at power generation.

Declarations

Conflict of Interest:

The authors declare that they have no conflict of interest.

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Figures

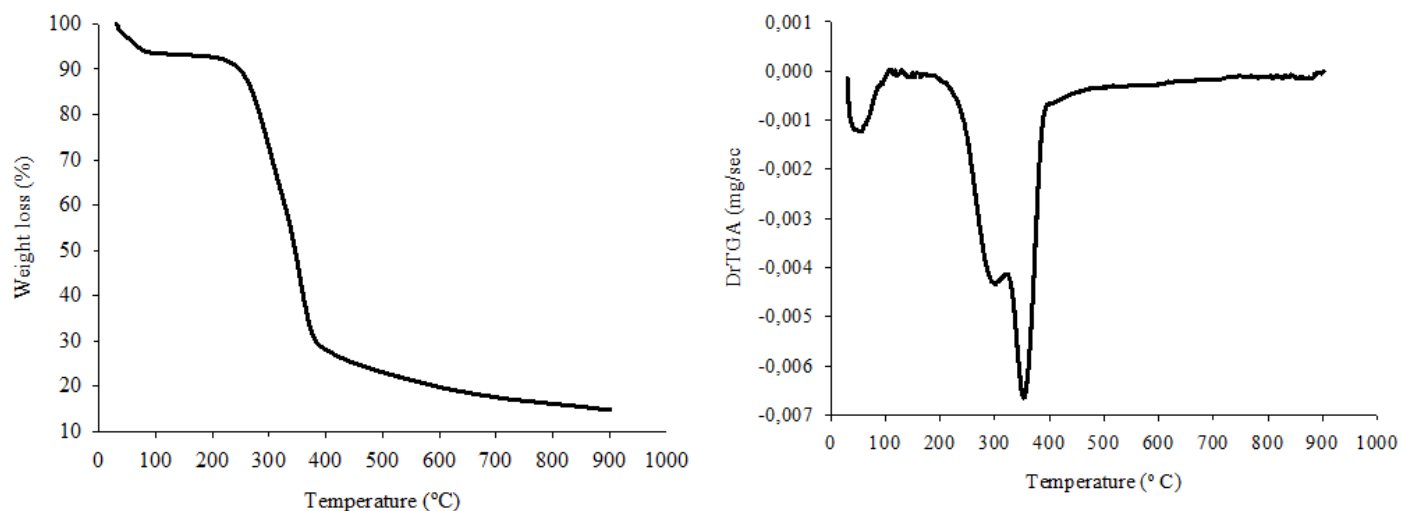


Figure 1

Thermogravimetric analysis and its derivative for baru biomass

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