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Characterization of pequi shell *(caryocar brasiliense camb.)* For its use as a biomass

Caracterização da casca de pequi *(caryocar brasiliense camb.)* Para sua utilização como biomassa

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ABSTRACT

Pequi is one of the most important fruits of Cerrado biome, and it is very consumed mainly by the population of west central of Brazil. The pequi is composed of bark, pulp and seeds, the bark of the ripe fruit correspond to approximately 84% of its weight, being the great majority discarded as garbage, causing environmental impact. On this, this research had as objective to characterize the barks of the pequi, aiming its use as biomass. For this purpose, moisture content analyzes were performed, and ashes, volatile materials, fixed carbon, specific mass, thermogravimetric analysis (TGA), grain size, and higher calorific value analysis as well, according to Brazilian standards ABNT NBR 8112 and 8633. The analyses showed that the bark of the in natura pequi has a high moisture content, around 73%, and 2.2% of ash content. The higher calorific value obtained to the pequi bark was around 15 MJ kg-1 being similar to other agricultural leavings used to generate electrical power. The thermogravimetric analysis showed that the pequi bark has a high percentage of lignin being about 50% compared to 30% in the eucalyptus bark, the higher the amount of lignin the higher the calorific value. The results obtained showed that the pequi bark presents high using potential as a biomass to generate electrical power.

Keywords: thermal energy; immediate analysis; TGA.

RESUMO

O pequi é uma das frutas mais importantes do bioma Cerrado, e é muito consumido principalmente pela população do Centro-Oeste do Brasil. O pequi é composto por casca, polpa e sementes, a casca do fruto maduro corresponde a aproximadamente 84% do seu peso, sendo que a grande maioria é descartada como lixo, causando impacto ambiental. Diante disto, esta pesquisa teve como objetivo caracterizar as cascas do pequi, visando sua utilização como biomassa. Com este propósito foram realizadas análises de teor de umidade; cinzas; materiais voláteis; carbono fixo, massa específica, análise termogravimétrica (TGA), granulometria e poder calorífico superior, segundo as normas brasileiras da ABNT NBR 8112 e 8633. As análises mostraram que a casca de pequi in natura apresentou um alto teor de umidade, em torno de 73 %, o teor de cinzas foi de 2,2%. O poder calorífico obtido para a casca do pequi foi entorno de 15 MJ Kg-1 sendo semelhante a outros resíduos agrícolas usados para geração de energia térmica. As analises termogravimétricas mostraram que a casca de pequi possui uma alta porcentagem de lignina sendo cerca de 50% comparado com 30% na casca do eucalipto, quando maior a quantidade de lignina maior o valor do calor específico. Os resultados obtidos mostraram que a casca de pequi apresenta elevado potencial de utilização como biomassa para geração de energia térmica.

Palavras-chave: energia térmica; análise imediata; TGA.

1 INTRODUCTION

The Cerrado biome is present in different Brazilian regions, occupying great extensions of the national territory, but is predominant in the west central region. It is formed by vegetation composed of sparse trees, shrubs and grasses, and among the most famous trees of the Cerrado biome is the pequi tree (RIBEIRO et al 2014). The pequi tree, belongs to the family *Caryocaraceae*, which produces pequi (*Caryocar brasiliense Camb.*). Pequi is widely used in cooking and in the agricultural industry for extraction of oils and production of liquors, pharmaceuticals (COLOMBO et al., 2015; PALMEIRA et al., 2016), being a kind of extractives economic base that feeds several families and serves as an alternative of income, both for rural and urban environments (MELO JUNIOR et al., 2012; SANTOS et al., 2013).

Pequi is composed of bark, pulp and seeds, the bark of the ripe fruit represent around 84% of the weight, the pulp represents 10% and the seeds 6% of the total weight. The medium weight of the pequi fruits is 339.3 ± 55.7 g; the medium length is 8.0 ± 0.3 cm; the medium width 8.8 ± 0.6 cm (LEÃO et al., 2017).

In the last years, many researchers developed in Brazil are searching different uses for the fruit of the pequi tree. Among these researches, some are aimed at the exploitation of the bark, giving it a noble destination. Being this the bigger part of the fruit, it generates a big quantity of wastes, that in its majority is discarded in non-appropriated locals, causing environmental pollution, at urban centers (MACHADO et al., 2015; LIMA et al., 2007; AMORIN et al., 2016; ROESLER et al., 2008).

According to Elwan et al. (2015), global waste generation in 2010 has increased much more than in the last ten years, along with population growth. A growth of around 3 billion of habitants are expected, generating 1.3 millions of tons of solid urban wastes per year, at a per capita rate of 1.2 Kg per person per day with the expectation of increase to 1.4 Kg per person per day in 2015. Thus, if the wastes generated by the consumption of the pequi fruit begin to be properly used, it creates a possibility to add economic value to the product, and avoid the increase of solid wastes at the landfills.

With the search for renewable sources of energy, biomass residues appear as an interesting option, valuing a residue an expense can be eliminated, in addition to providing an extra income for the generate industry, adding value to the reduction.

Cardoso et al. (2015) and Miranda et al. (2013), studying agricultural residues claims to be of great importance the knowledge of the thermal properties of vegetal residues, since they are associated to applications such as the generation of energy from biomass, thus helping in their use. Knowledge about flame retardant cellulosic fibers gives us information about the combustion process for the production of nanocellulose that can be a source of nanoparticles with application in polymers.

Combustion is used to chemically convert energy stored in the biomass into heat (GUMISIRIZA et al., 2017). To determine the potentiality of the combustion of a fuel and to evaluate its best application, one must first know its fundamental characteristics (chemical and physical), i.e., moisture content, elemental composition, immediate composition and calorific power (HAYKIRI-AÇMA, 2003).

The degradation processes, such as thermal stability, weight loss, biomass composition, thermal capacity, phase changes and some reaction that occurs with the substrate, has been studied by thermogravimetric techniques such as TGA and DSC. These techniques help in identifying the amount of water in a material, which can be prejudicial in some cases. The information about weight loss may lead to the knowledge of the percentage composition of the sample components, which favors the use of many materials such as biomass (GONÇALVES et al., 2009).

When the biomass is used to generate heat, one of the most important properties that has to be controlled is the moisture content, since the lower the moisture content, the higher the heat production per unit mass. However, the ash content in large quantities, above 5%, for being composed by salts, can cause serious damages to the equipment that are used to carry out the combustion (LIU et al., 2017).

Therefore, every time that a biomass is used as fuel, it is necessary a knowledge about the chemical and physical properties, because this will influence directly in the quality of the fuel and in the way that will be realized the combustion. With the previously exposed, this research is intended to verify the viability of the technic of exploitation of the pequi barks to energetic purposes, in process of thermal conversion.

2 MATERIAL AND METHODS

2.1 MATERIAL

Pequi barks were obtained in Anápolis-GO open fairs in January of 2014, and 5 pieces of eucalyptus were obtained at a company that sells firewood to combustion in furnace at Anápolis commerce. Posteriorly the eucalyptus pieces were ground and the sawdust were collected and sifted in a 4 mm sieve to posterior evaluation of weight loss in TG and comparations to the pequi, being the eucalyptus wood more studied as energy source.

Five fresh samples of pequi were obtained (*Caryocar brasiliense Camb.*), with around 4 kg each. Posteriorly it was placed in a plastic package and taken to the lab, then the samples were unpacked and placed in the same container and were homogenized, then, 3 samples of around 50 g were removed, to determine the initial water content. The barks were placed at a laboratory stove with forced circulation at a temperature of 35 ± 5 °C, until the final water content is close to 10% b.u.

Next, the pequi barks were crushed and sieved, collecting only the particles that passed through the nº 5 (4 mm) sieve, and then they were placed in plastic containers and stored in the cold room for further physical and chemical analyzes.

2.2 FINAL WATER CONTENT DETERMINATION

To determine the water content of the pequi biomass, 3 samples were collected, having around 50 g, and were placed at a laboratory stove at 100 ±10 ºC, with forced circulation until obtaining constant weight, according to standardization of the Brazilian Association of Technical Standards (ABNT), by the norm NBR 8112/86 (ABNT, 1986).

2.3 DETERMINATION OF APPARENT SPECIFIC MASS

Apparent specific mass of the biomass represents the quantity of biomass that occupies one unity of volume, it was determined by means of a balance with a cylinder of volume and mass known, where the cylinder was of biomass, then weighted again obtaining the mass of biomass in a known volume, being realized three repetitions to each biomass, and the results were expressed in kg m^{-3}.

2.4 DETERMINATION OF CALORIFIC VALUE

Calorific value indicates how many calories can be obtained from a material when it suffers the combustion process, that is, it is an indicator of quality of biomass to heat generation. The calorific value can be superior or inferior, but the superior calorific value will always be higher, due to taking into account the latent heat of condensation of the fuel water of constitution.

2.5 SUPERIOR CALORIFIC VALUE (SCV)

To verify the influence of humidity on calorific value, 5 samples with known water content were separated, and were then humidified to reach the final water content of about 10, 20, 30, 40 and 50% bu.

Superior calorific value of the pequi bark to samples with different moisture content was determined according to the norms of the American Society for Testing and Materials – ASTM (1977) D-2015-66, by means of an isothermal calorimeter pump model E2K. The samples were of around 0.5 g of biomass, each one was placed at a cylinder and then the cylinder with the sample was placed in the calorimetric pump. Initially the set (cylinder-sample) received gradually 30 atm of oxygen in the calorimetric pump, and after that procedure, the pump was coupled to the calorimeter that gave the ignition automatically, and after a few minutes, the calorific value was quantified.

2.6 INFERIOR CALORIFIC VALUE (ICV)

Inferior calorific value was obtained indirectly, using the methodology cited by Leite et al. (2014), through Equation (1):

$$\text{ICV}=\text{SCV}\left(\frac{600 \text{ x } 9\text{H}}{100}\right) \qquad (1)$$

Where: ICV = inferior calorific value (kcal/Kg); SCV = superior calorific value (kcal/Kg); H = hydrogen content (%); 600 = heat of condensation of water at 0 °C; 9 = Represents the amount of water (in kilograms) that form by oxidizing one kilogram of hydrogen.

2.7 GRANULOMETRY

To obtain the biomass granulometry, it was added in sieve sizes ranging from 4 mm or 5 Mesh to 0.075 mm or 200 Mesh of the Bertel brand, being agitated for 40 minutes in a mechanical stirrer. Subsequently the material retained in each sieve was weighed obtaining the retained weight accumulated as well as the retained percentage accumulated.

2.8 CHEMICAL PROPERTIES ANALYSIS

The immediate analyzes of the biomass were realized according to the standardized method by Brazilian Association of Technical Standards (ABNT), by the norm NBR 8112/86 for: ash content (AC), volatile solids content (VSC) and fixed carbon (FC) (ABNT, 1986). Subsequently, the elemental composition was evaluated according to the methodology of Parikh et al. (2007).

2.9 ASH CONTENT

The ash content was determined using three samples of about 1g, that were dried in a laboratory stove at 103 ± 2°C until reach constant weight. After the drying, the samples were taken to the muffle furnace at 650±10 °C for six hours. Next, the samples were cooled in desiccator until reach room temperature, the ash content was determined by the difference of the initial and final mass.

2.10 VOLATILE SOLIDS CONTENT

The percentage of volatile material determines the quantity of gaseous products, without the presence of the moisture that is released from the sample under specific conditions. The samples, having around 1 g, were previously dried in laboratory stove at 103±2 °C until reach constant weight. Then it was taken to muffle furnace at 950±10 °C for nine minutes. After this procedure the samples were removed from the muffle furnace, cooled in desiccator until reach room temperature, and then the volatile solids content was determined by weight difference.

2.11 FIXED CARBON

The fixed carbon represents the amount of carbon chemically bonded to another carbon. This is a result of the sum of the percentages of moisture, ash and volatile material, subtracted from 100 according to ABNT 8112/86.

2.12 ELEMENTAL COMPOSITION

To determine the elemental composition (Carbon-C, Hydrogen-H, and Oxygen-O) it was used the methodology proposed by Parikh et al. (2007), where it takes into consideration the values of fixed carbon (CF) and volatile solids content (VSC), as observed in Equations 2, 3 and 4.

$$\mathrm{C}(\%) = 0.637(\mathrm{CF}) + 0.455(\mathrm{VSC}) \tag{2}$$

$$\mathrm{H}(\%) = 0.052(\mathrm{CF}) + 0.062(\mathrm{VSC}) \tag{3}$$

$$\mathrm{O}(\%) = 0.304(\mathrm{CF}) + 0.476(\mathrm{VSC}) \tag{4}$$

Where: C= Carbon percentage (%); H= Hydrogen percentage (%); O= Oxygen percentage (%); CF= Fixed carbon; VSC= Volatile solids.

2.13 THERMOGRAVIMETRIC ANALYSIS (TG/DTG)

The data were obtained using a TG/DTG of Perkin Elmer. The samples' mass were of 6.492 mg for pequi and 5.926 mg for eucalyptus, with a rate heating of 10 ^{o}C min^{-1}, in oxygen and nitrogen atmosphere, with a flow rate of 20 mL min^{-1} and a heating range of 30 to 900 ^{o}C for oxygen and nitrogen. The samples of eucalyptus were used as a standard for comparation with pequi and literature data (PEREIRA et al., 2013).

3 RESULTS AND DISCUSSION

The granulometry of the crushed pequi bark was determined and the data is exposed in Table 1, which will be used to the physical-chemical analysis. It is observed in this Table that the sample possessed particles between 2 to 0.15 mm, being that the majority of the samples were bigger than 1.00 mm. According to Vieira et al. (2013) small particles below 0.15 mm (practically dust) can negatively influence in the combustion process, because the oxygen does not mix uniformly with the biomass, interfering in the physical-chemical characterization of the same.

TABLE 1: Granulometric analysis of the pequi bark samples used to perform characterization

Sieves n.	Diameter	Weight Withheld Accumulated (g)	Retained Accumulated (%)	Accumulated Pass (%)
5	4.00	0.00	0.00	100.00
10	2.00	1.64	0.78	99.22
18	1.00	112.97	53.55	46.45
25	0.71	154.05	73.02	26.98
50	0.30	187.86	89.05	10.95

100	0.15	205.31	97.32	2.68
200	0.08	210.96	100.00	0.00

The Table 2 shows the results found to the physical-chemical properties, (physical, immediate analysis and elemental composition) to the pequi bark. It can observe that the average initial water content of the samples were around 73%, that is considered high when the material is intended for burning, but this humidity values found were similar to the values of the initial water content found for the cocoa husks (PEREIRA et al., 2013). According to Vale et al. (2011) the moisture content is a factor of great importance in the use of biomass as fuel, because it influences on the capacity of generating heat during the combustion process.

According to Vieira (2013) the ashes are generally constituted of silicon compounds (Si), potassium (K), sodium (Na), sulfur (S), calcium (Ca), phosphorus (P), magnesium (Mg) and iron (Fe), being obtained by resulting residues from the combustion of organic components and oxidation of inorganics, it can vary in function of the biomass used in the burning.

> ***The knowledge of ash content will directly influence in the process of incineration, just as in temperature and in incineration time of a biomass. The pequi bark presented ash content around 2%, similar values was found to other biomass like araucaria sawdust, pinus from commercial plantation (SCHMATZ et al., 2016) and pericarp of macaúba (EVARISTO et al., 2015).***

According McKendry (2002) the volatile content is quantified measuring the mass fraction of the biomass that volatilizes during the heating of a standard sample previously dried in an inert atmosphere to temperatures of approximately 850 °C. The volatile content found was of 85.26%, similar to the one found for the sugarcane bagasse, that was 79.70% and 85.61% found by Santos et al. (2011) and Paula et al. (2011), being one of the most used at the present time. The biomass that presents high volatile content has greater ease of ignition in the combustion process, although this process becomes difficult to control (VIERIA et al., 2013).

It is verified in Table 2 that the value of fixed carbon content was 16.93% similar values were found by Dermibas (2004) for different biomass. The fixed carbon content of a biomass influences in the amount of heat generated, and it is determinant in the time of burning of the same, that is, the higher of this percentage the slower the fuel will burn, it is defined as the remaining mass without the moisture, the volatile compounds and excluding the ashes (VIEIRA et al., 2013).

The elementary analysis of the pequi bark presented promising results for its use as a biomass and thermal energy generation, since the percentage of carbon obtained was of 48.11%, relatively higher than that of oxygen, that was of 45.73% and around 8 times higher than the hydrogen that was of 6.16%, which means a greater amount of energy released in the combustion. And the values found in the elementary analysis to the pequi bark were similar to the values found by Paula et al. (2011) to sugarcane bagasse.

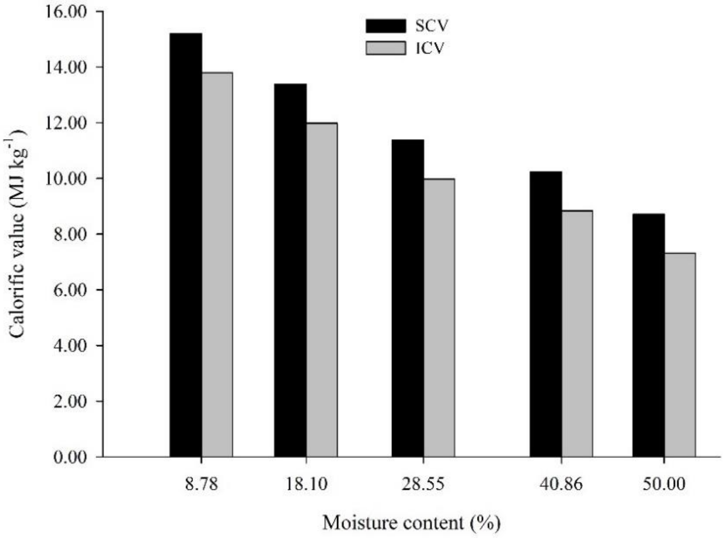
TABLE 2: Mean values found in the physic-chemical characterization of the pequi shell samples collected in January 2014

Variables analyzed		Mean values
Physical properties	Initial Moisture (%)	73. 60
	Final Moisture (%)	09.50
	Apparent specific mass (kg/m^3)	577.55
Immediate analysis	Ash (%)	02.19
	Volatile Solids (%)	85.26
	Fixed carbon (%)	16.93
Elementary composition	Carbon (%)	48.11
	Hydrogen (%)	06.16
	Oxygen (%)	45.73

The figure 1 shows the average value of superior calorific power (SCV) and inferior (ICV), obtained to samples with different moisture content. According to Leite et al. (2014) the SCV and the ICV of a determined biomass are the most important physicochemical properties to be considered for the choice of a thermochemical process, because they indicate the quantity of energy released during the transfer of the heat, related to the process efficiency, that is, the higher the SCV, more efficient is the biomass to generate thermal energy (VIEIRA et al., 2013).

The maximum average values found to the SCV were 15.21 MJ kg^{-1} with 9.5% of humidity, the average values to the ICV were 8.46 MJ kg^{-1} to 50% of water content. Similar values were found by Santos et al. (2011) using different clones of eucalyptus, and by Vieira et al. (2013) too, using rice husk. It is observed that the moisture exerts great influence in the calorific power, the lower the humidity the higher the calorific power. This was confirmed by Calegari et al. (2005).

FIGURE 1**:** Results obtained to superior calorific power (SCV) and inferior (ICV) as a function of moisture content



Thermogravimetric analysis allow the verification of the stages of weight loss in function of the temperature, this technique allows the evaluation of the release of volatile compounds, humidity and events like chemical and physical reactions, all these properties influence in the utilization of the material to the energy production and are directly related to the calorific power. Thermogravimetric curves (TG) and their derivatives (DTG) were obtained to the pequi samples, and eucalyptus was used as a comparison standard in an oxygen oxidizing atmosphere and nitrogen inert with a heating rate of 10 ^{o}C min^{-1},the results are presented in the figures 2 and 3, respectively. The curves of DTG show the maximum peak of temperature to degradation (Figures 2 and 3).

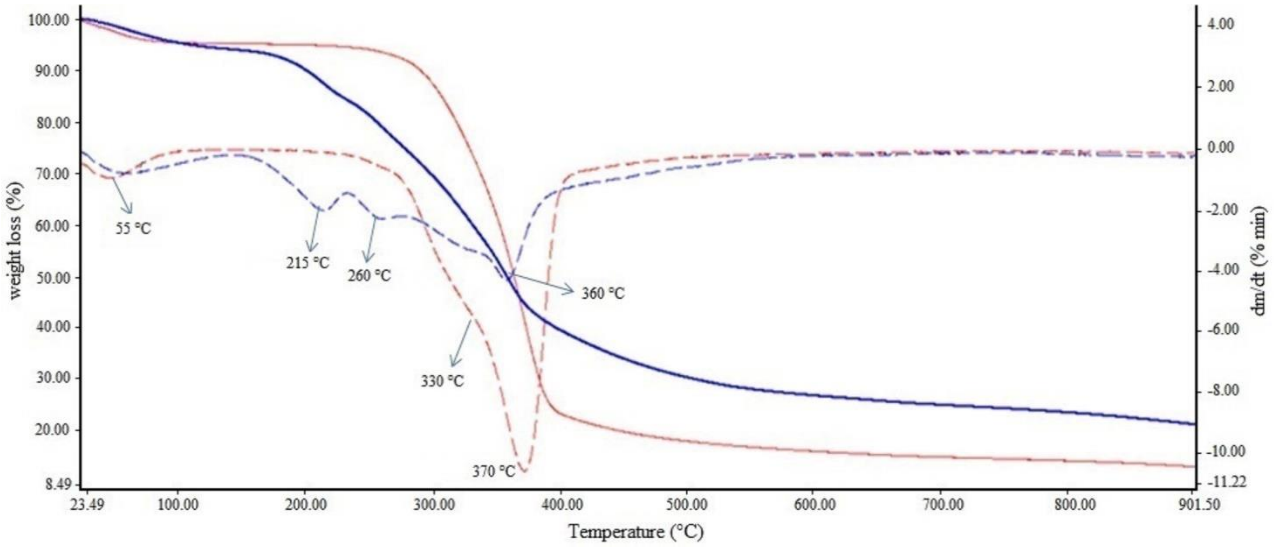
Some works about the thermogravimetric analysis of wood report that the first stage of loss mass occurs in temperatures lower than 100 °C and is related to the loss of moisture. The second and third stages, which happens in a temperature between 200-450 °C, can be related to the burning of organic matter, which are hemicellulose and cellulose, above 450 °C the burning of the remaining lignin occurs. The lignin presents degradation throughout the heating range from 100 to 900 °C, can be decomposed together with cellulose and hemicellulose, but there are no characteristic peaks of its degradation in the DTG. The fact that lignin decomposes in a wide range is due to its structure, that have three-dimensional, amorphous and branched macromolecules, and consists of basic units of phenyl propane (GIUDICIANNI et al., 2013; SANTOS et al., 2011).

The TG/DTG curves to the eucalyptus sample shows 3 stages of weight loss, and the second stage is not well defined, these data are known and observed in literature. Pereira et al. (2013) used the bark of 6 eucalyptus clones and analyzed the weight loss in an atmosphere of nitrogen at a heating rate of 10 °C min^{-1}, and states that the first average weight loss is 6.47% due to the drying of the biomass. The second stage from 250 to 300 °C has an average weight loss of 17.42% related to the degradation of hemicelluloses, and the presence of a horizontal plateau in the eucalyptus thermogram shows that these compounds are more stable. The weight loss of 32.09 and 15.83% in average, totalizing 47.93%, are related to the degradation of hemicelluloses and cellulose, which occurs in temperature ranges of 300-350 °C and 350-400 °C, because the structure of the cellulose presents monomers, the degradation requires a greater amount of energy for the depolymerization of the cellulose chain. These data may differ according to the species of the plant. It is observed in the two atmospheres (Figure 2 and 3) that the hemicellulose in the eucalyptus bark is more stable when compared to pequi, since the beginning of the degradation occurs at higher temperatures.

The figure 2 presents the TG/DTG curves to the pequi bark and eucalyptus under nitrogen atmosphere at a heating rate of 10 °C min^{-1} obtained for comparison. To the eucalyptus curve presented in the graphic of Figure 2, values of weight loss were obtained to the 3 stages of 4, 23, 70 and 46.49%, which presented similarity with the ones obtained by Pereira et. al. (2013), being observed a total of 70.19% of organic matter decomposition, constituted of hemicellulose and cellulose, and about 30% of remaining lignin. Since eucalyptus bark wood is widely used for energy generation (SANTOS et al., 2011).

To the pequi, in the same atmosphere used, that is, nitrogen, it is observed that the second stage of weight loss is more defined being observed 5 stages, like it is showed by Figure 2 and DTG curve. The first stage with 4.62% of weight loss is related to the loss of humidity in temperatures lower than 100 °C and possible loss of volatile compounds, the second and third stages of 200-300 °C are related to the loss of hemicelluloses with weight loss of 8.04 and 10.24%, totalizing 18.28%, the fourth stage of 300-400 °C with weight loss of 31.98% is related to the burn of the cellulose and the fifth stage above 400 °C is related to the burning of the remaining lignin. The difference in the profiles of the thermogravimetric curves of pequi and eucalyptus is related to differences in chemical composition and thermal stability. For the pequi a total of 50.26% of hemicellulose and cellulose was observed, this shows that the pequi has a greater amount of lignin and a somewhat greater stability in the burning of lignin, about 50% remaining, the higher the amount of lignin the higher the calorific value. Santos et al. (2011) points out that the presence of lignin is extremely important for the production of charcoal, the higher the lignin content the greater the conversion to charcoal and the thermal stability is related to the presence of more condensed structures.

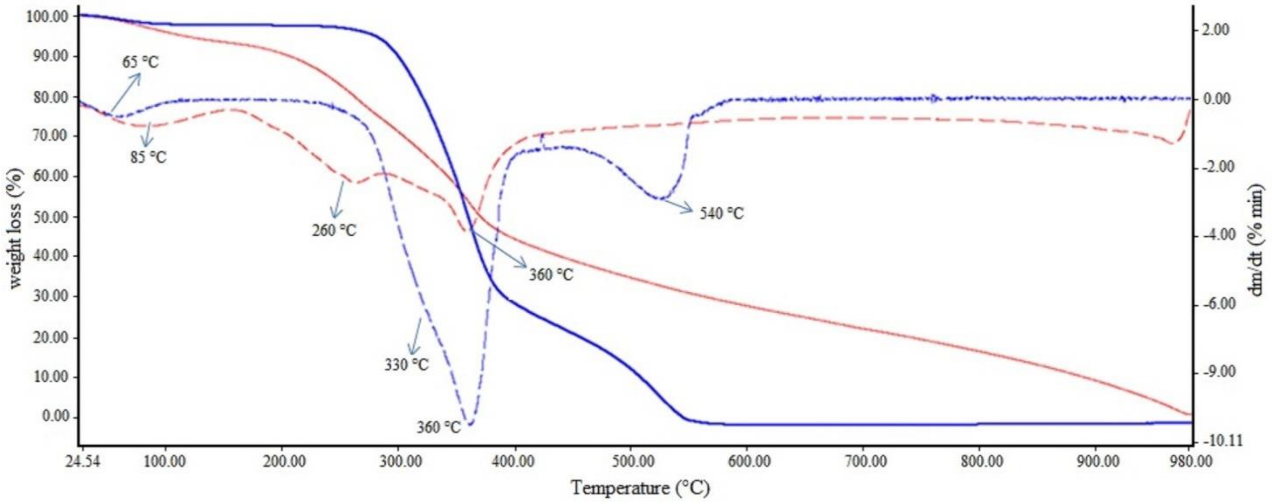
FIGURE 2: Curves of TG/DTA to the pequi (blue) and eucalyptus (red) samples, analyzed dry and ground after collection, obtained in nitrogen atmosphere and, at a heating rate of 10 °C min^{-1}



In oxygen atmosphere, the TG/DTG curve shows a slightly different behavior. To the eucalyptus the stages of weight loss are also 3, being that the second stage is not well defined and a well-defined stage is observed in this case to the burning of lignin of 400-600 °C. The weight loss percentages obtained to the eucalyptus were of 1.55% to the first stage related to the humidity loss in temperatures lower than 100 °C, 63.6% on the second to the hemicellulose and cellulose burn with temperatures of 200-400 °C and 34.7% on the last stage to the lignin burn.

To the pequi, on the first stage is observed a weight loss of 3.5%, to the second and third stages it is observed a weight loss of 15.7 and 23.9% totalizing 39.6% to the burn of hemicellulose and cellulose, and the final stage of lignin burn starts and a higher percentage and extends to higher temperatures, showing that the pequi has more lignin in its composition, totalizing 60.4% of weight loss.

FIGURE 3: Curves of TG/DTA to the pequi (blue) and eucalypt (red) samples, analyzed dry and ground after collection, obtained in oxygen atmosphere and, at a heating rate of 10 °C min^{-1}.



4 CONCLUSIONS

Pequi bark shows potential to be used as an energy input for thermal energy generation through the direct combustion process, but showed a high initial water content requiring a drying process.

The volatile solids contents were relatively high and the ash content low, which allows pequi bark to be considered biomass for combustion. On the other hand, the elemental analysis of the biomass was propitious, since the percentage of carbon obtained was relatively higher in relation to that of oxygen and that of hydrogen, which means a greater amount of energy.

The thermogravimetric analysis showed to be an important tool to verify the loss of mass, contributing significantly to characterize the combustion temperatures of the chemical composition of the same.

The higher calorific value found was similar to those of some biomasses used today, in combustion processes such as sugarcane bagasse and eucalypt, and thus, pequi bark shows itself as a promising biomass.

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