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KINETIC PARAMETERS AND COMBUSTION THERMAL BEHAVIOR OF AÇAÍ FIBROUS SEED BY THERMOGRAVIMETRIC ANALYSIS

Fernando Henrique de Barbosa dos Santos
Federal University of Pará, Av. Augusto Corrêa 1, LABEM, 66075-110 Belém-PA.
Fernandohenrique13@yahoo.com.br

Alan Nogueira Carneiro
Federal University of Pará, Av. Augusto Corrêa 1, LABEM, 66075-110 Belém-PA.
Alancarneiro90@hotmail.com

Ana Paula Mattos
Federal University of Pará, Av. Augusto Corrêa 1, LABEM, 66075-110 Belém-PA.
mattos.ufpa@gmail.com

Danielle Regina da Silva Guerra
Federal University of Pará, Av. Augusto Corrêa 1, LABEM, 66075-110 Belém-PA.
daguerra@ufpa.br

Manoel Fernandes Martins Nogueira
Federal University of Pará, Av. Augusto Corrêa 1, LABEM, 66075-110 Belém-PA.
mfmn@ufpa.br

Abstract. Açaí fruit production in 2019 reached about 1.4 million tons in Pará, of which about 70% is composed of the so-called fibrous seed. The importance of the fruit in the Amazon region makes the fibrous seed waste a potential renewable fuel, mainly when applied in co-combustion processes with coal. For such an application, it is necessary to know the thermal behavior of the residue during combustion by kinetic parameters and mass loss curves so that adjustments can be made to energy conversion equipment. This work aims to evaluate the combustion kinetics of the fibrous seed waste by thermogravimetric analysis (TGA) and Arrhenius parameters obtained by the Kissinger-Akahira-Sunose (KAS) and Flynn-Wall-Ozawa (FWO) methods. Samples of bituminous coal and açai seed with fiber and without fiber were heated from 298 to 1073 K in an O_2 atmosphere at 40 ml.min^{-1} at three heating rates: 10, 20, and 30 K.min^{-1}. The mass loss (TG) and mass loss rate (DTG) curves allowed the identification of two stages of combustion. The first was related to the release of volatiles and the second to char oxidation. The peaks of higher mass loss rate for the first stage vary between 554-569 K, while the second stage varies between 676-729 K. The results obtained by the KAS and FWO methods showed slight variation between each other. The activation energy varies between 119.1-168.7 kJ.kmol^{-1} (coal), 186.1-301.5 kJ.kmol^{-1} (seed with fiber) and 170.6-1161.8 kJ.kmol^{-1} (seed without fiber). The variation of the activation energy indicates that the reaction mechanism for the burning of açaí is not the same for the entire combustion process, dependent on the degree of conversion, unlike coal whose values show slight variation as a function of conversion degree. The açaí seed presents high peaks of mass loss rate during the release of volatiles, which suggests the formation of more intense flames during the initial burning stages in comparison to coal, which has no related peaks related to the release of volatiles.

Keywords: biomass, açaí seed, isoconversional methods, combustion, thermogravimetry

1. INTRODUCTION

In the context of bioeconomy and sustainability, more effective initiatives are need for the development and use of renewable energy sources that reduce the environmental problems. Brazil stands out in the global scenario by presenting 47.4% of its energy matrix composed of renewable sources in the domestic energy supply (DES) which sugarcane biomass represents 15.4%, Figure 1, while other biomasses, like rice husk and vegetable oils, have a participation about 19.1% of the total 7% of "other renewable sources": biodiesel, other biomass and biogas (MME, 2023).

It is clear that in national context other biomasses have a less participation in the energy matrix compared to sugarcane biomass. When evaluated in the economic region which it is located, as is the case of the açaí fruit production in the Amazon region, they have some advantages in an economical and environmental ways: lower greenhouse gases emissions, like SO_2 e NO_x (Macile-Silva et al., 2019), the residues insertion and valorization in the local economy (Brandão et al., 2021).
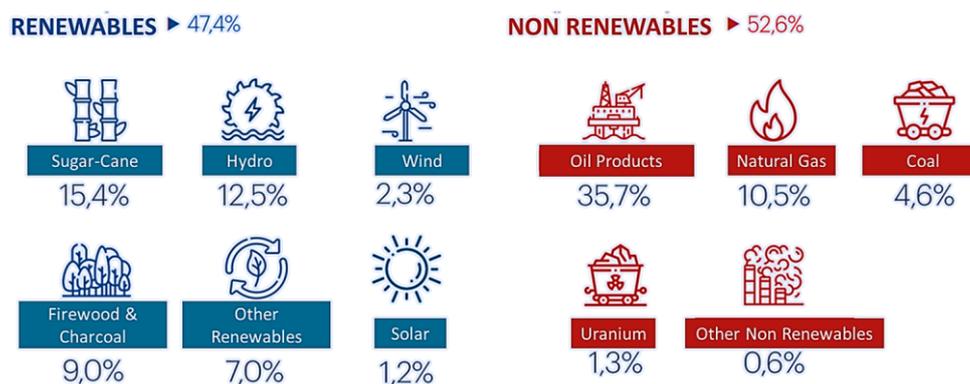
Figure 1. Brazil domestic energy supply (DES) (Adapted from MME, 2023).

The açaí fruit production in the Pará state reached about 1.4 million tons in 2019 and when evaluating the residue generation about 70% is composed of the so-called "fibrous seed", that is the seed of the fruit after extraction of the açái pulp (SENAI CETIQT, 2021). The açaí fibrous seed application in energy conversion in the Pará state becomes a plausible alternative. Biomass residues also have some issues: high moisture content, low heating value, heterogeneous composition and properties (Araujo et al., 2022).

The correct application of fibrous seed in combustion processes requires data about its physic-chemical properties, such as its elemental composition and the combustion kinetics parameters. The equipment design for biomass combustion requires a good knowledge of such properties (Yorulmaz et al., 2009) besides ensuring the development of techniques for the correct management of the material, such as transport and storage (Liu et al., 2021).

Kinetic parameters data has been study for numerous biomass whose main methodology is known as "Model-Free" or "Isoconversional" that allows the calculation of activation energy in a simple way and avoiding errors associated with the choice of mechanism or reaction model (Slopiecka et al., 2012). Such methods were evaluated from data obtained by thermogravimetric analysis (TGA), which allow understanding the physic-chemical properties of a biomass in combustion processes, such as its reactivity (Rico et al., 2022).

In this context, the objective of this work is to present the combustion kinetic parameters, more expecificy the activation energy, for açaí fibrous seed estimated from the following isoconversional methods: Flynn-Wall-Ozawa (FWO) and Kissinger-Akahira-Sunose (KAS). Those methods are the most widely used in chemical kinetics research for estimate the activation energy (Vyazovkin et al., 2011) and require mass loss (TG) and mass loss rate (DTG) data obtained by thermogravimetric analysis (TGA) at least three heating rates (Colpani et al., 2022).

2. KINETIC THEORY

Biomasses are essentially heterogeneous fuels composed by hemicellulose, cellulose and lignin. Each component has a temperature range in which decomposition occurs: 493-588°C for hemicellulose, 588-673°C for cellulose and 433-1173°C for lignin (Yang et al., 2007). It is understandable that such residue presents a complex thermal behavior because of the interaction between those components and their respective concentrations.

The evaluation of kinetics parameters using "Isoconversional" methods allows the calculation of the activity energy without the need to determine a reaction model to describe the material. The method allows simplifying the chemical process to a global one-step reaction. Biomass residue have their thermal decomposition represented by Eq. (1) (Rahib et al., 2020):

$$Biomass\ (solid) \rightarrow Volátiles\ (gas) + Char\ (solid) \tag{1}$$

In order to describe the combustion reaction, the conversion degree of the material is calculated as a function of mass loss (TG) data as a function of time obtained by TGA. The conversion degree (α) of the material, represented by Eq. (2), represents the fraction of matter that was converted during heating whose values range from 0 to 1. The terms m_o, m_f and m_t are the initial, final and mass at time t, respectively.

$$\alpha = \frac{m_o - m_t}{m_o - m_f} \tag{2}$$

The chemical reaction rate, represented by Eq. (3) is a function of the reaction rate constant k and the reaction model function f(α), and is dependent on the degree of conversion. The reaction rate constant is described by the Arrhenius equation, Eq. (4), and relates the rate constant as function of the temperature T(K), the pre-exponential factor A(s^{-1}), the activation energy E_a(kJ.kmol^{-1}), and the universal gas constant R (8.314 kJ.kmol^{-1}.K^{-1}).

$$\frac{d\alpha}{dt} = k(T).f(\alpha) \tag{3}$$

$$k(T) = A.e^{-E_a/R.T} \tag{4}$$

Thermogravimetric analysis for non-isothermal methods is performed with a constant heating rate (β), Eq. (5), so equation 3 can be modified by taking the heating rate and Arrhenius equation into account, represented by Eq. (6).

$$\beta = \frac{dT}{dt} \tag{5}$$

$$\frac{d\alpha}{dT} = \beta^{-1}.A.\exp(\frac{-E_a}{R.T}).f(\alpha) \tag{6}$$

The reaction models f(α), as discussed by Vyazovkin et al., (2011), are mostly specified for solid state reactions, as is the case of biomass residue. The author also addresses that even though the method does not require knowledge about the reaction model, however the resolution of Eq. (6) must still obey some model, thus the choice of the model is crucial for solving kinetic methods. Equation 7 is the integral form of the reaction model f(α), which is essential for the solutions of kinetic problems.

$$g(\alpha) = \int_0^\alpha \frac{1}{f(\alpha)} d\alpha = \beta^{-1}.A.\int_{T_0}^T \exp\left(\frac{E_a}{R.T}\right) dT \tag{7}$$

3. MATERIALS AND METHODS

3.1 Sampling and characterization

Açaí fibrous seed was obtained from companies that collect and manage the disposal of such material in the region of Igarapé-Mirin\Pa. Two types of seeds were analyzed: seeds with fiber (residue without any pre-treatment) and seeds without fiber (residue with pre-treatment to remove fibers and clean the material). In order to characterize the material in terms of co-combustion, a bituminous coal sample was analyze together with the açaí fibrous seed.

The samples were submitted to the following processes for standardization to the energy characterization analyses: drying process according to the standards NBR-16508 (coal) and CEN/TS 14774-1 (solid biofuels), grinding process in a bench knife mill and standardization of the dimensions in sieves between 30-60 mesh.

The following analyses were performed for the fuel characterization in terms of combustion process: proximate analysis, higher heating value (HHV) analysis and thermogravimetric analysis (TGA). Figure 2 represents the samples used in this work: bituminous coal, acai seed with fiber and açaí seed without fiber.
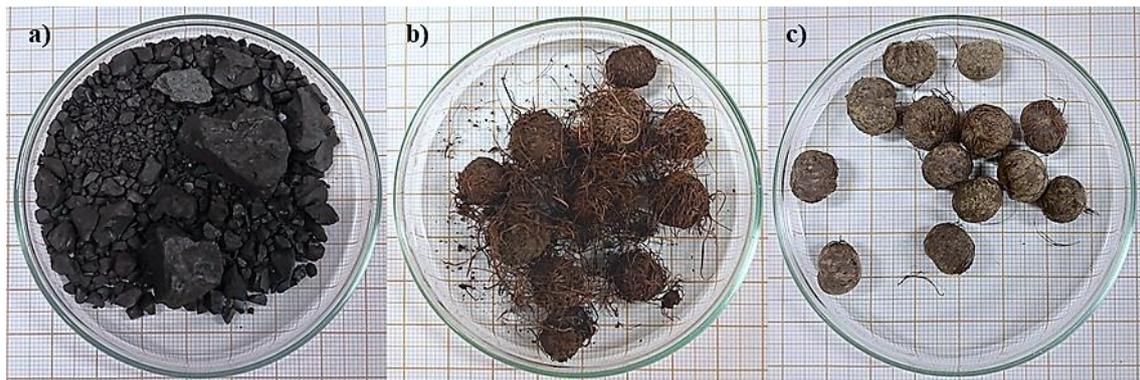

Figure 2. Samples: a) bituminous coal, b) açaí seed with fiber and c) açaí seed without fiber.

3.2 Characterization analysis

Biomass residues need to be characterized from an energy point of view for their evaluation as a fuel. The proximate analysis aims to determine the contents of moisture, volatile matter, fixed carbon and ash and was evaluated according to the standards for coal (NBR 16587 and 16586) and for solid fuels in general, such as biomass (CEN/TS 15148 and 14775). For comparison purposes, the contents were evaluated on a dry basis.

The higher heating value (HHV), which represents the amount of energy released by the fuel during the combustion process, was obtained automatically by means of a calorimetric pump, model C 2000 Ika Werke. All analyses were performed in triplicate and the average value for each material was evaluated.

3.3 Thermogravmetric analysis (TGA)

Thermogravimetric analysis was performed on a vacuum-sealed thermo-microbalance, Libra-Netzsch, TG 209 F1. The analysis generates mass loss (TG) and mass loss rate (DTG) data, essential for describing the thermal behavior of the material during a combustion reaction by identifying at what temperature mass loss reactions will occur.

The procedure was performed with the following parameters: heating from 298 to 1073 K, heating rates of 10, 20 and 30 K.min^{-1}, oxidizing atmosphere of O_2 with a volumetric flow rate of 40 ml.min^{-1} and sample mass of 10 mg.

The DTG curves allow the identification of the reactivity regions of the sample and through the software "Phroteus Thermal Analysis", integrated in the TGA equipment, the data treatment was performed to obtain the following information: higher mass loss rate (DTG peak), onset, endset and DTG peak temperatures for identification of combustion stages.

3.4 Isoconversional Method

The isoconversional methods used in this work, Flynn-Wall-Ozawa (FWO) and Kissinger-Akahira-Sunose (KAS), are based on the principle of solving the Eq. (7) and both assume a one-step reaction (Rahib et al., 2020). The methods present a linear solution since there is no analytical solution, so a difference between the results obtained is expected (Vyazovkin et al., 2011).

The expression developed by the Flynn-Wall-Ozawa (FWO) method, Eq. (8), allows the calculation of the activation energy through the linear relationship between various heating rates for the same degree of conversion by generating a graph $\ln(\beta)$ versus $1/T_\alpha$, where T_α represents the temperature relative to the degree of conversion. The activation energy is obtained using the slope angle of the linear approximation, $1.052(E_a/R)$.

$$\ln(\beta) = \ln\left(\frac{A.E_a}{R.g(\alpha)}\right) - 5.331 - 1.052\left(\frac{E_a}{R.T_\alpha}\right) \tag{8}$$

The Kissinger-Akahira-Sunose (KAS) method, Eq. (9), allows the calculation of the activation energy by generating the graph $\ln(\beta/T_\alpha{}^2)$ versus $1/T_\alpha$, where T_α represents the temperature relative to the degree of conversion. The activation energy is obtained by means of the slope angle of the linear approximation, E_a/R.

$$\ln(\beta/T_\alpha{}^2) = \ln\left(\frac{A.R}{E_a.g(\alpha)}\right) - \left(\frac{E_a}{R.T_\alpha}\right) \tag{9}$$

Both methods need a conversion degree value to perform the calculation. The range of values used varies from 0.05-0.95 with a step of 0.05, as recommended by Vyazovkin et al., (2011), in order to have a good representation of the variation of activation energy as a function of conversion degree.

4. RESULTS

4.1 Characterization Analysis

Table 1 shows de data of higher heating value and proximate analysis for the fuels in dry basis. The açaí seed samples presented a volatiles content of 77.6% (seed with fiber) and 78.2% (seed without fiber), higher in comparison to coal, 34.9%. Biomass residues in general have a high content of volatiles, such as the empty fruit bunch, 73.5%, and the mesocarp fiber of the oil palm fruit, 72.8% (Talero et al., 2019). A high volatile content suggests that the ignition of the açaí seed occurs at low temperatures (Santos et al. 2020).

Table 1. Proximate analysis and higher heating value (HHV) data.

Samples	Proximate Analysis (%. Dry Basis)			HHV (MJ.kg^{-1})
	Volatiles	Fixed Carbon	Ash	
Bituminous Coal	34.9 ± 0.19	49.7 ± 0.69	15.4 ± 0.53	26.8 ± 0.16
Seed With Fiber	77.6 ± 0.73	21.6 ± 0.78	0.8 ± 0.05	19.5 ± 0.09
Seed Without Fiber	78.2 ± 0.08	20.3 ± 0.12	1.5 ±0.05	19.3 ± 0.29

The açaí samples presented fixed carbon content of 21.6% (seed with fiber) and 20.3% (seed without fiber), whose values are in the range of 15-25%, which according to Hansted et al., (2018) can help in increasing efficiency in power generation processes given the slow burning of the solid phase.

As observed in table 1, the coal sample presented a higher HHV, 26.8MJ.kg^{-1}, while the açaí seed samples presented values close to each other, 19.5MJ.kg^{-1} (seed with fiber) and 19.3MJ.kg^{-1} (seed without fiber), values similar those found in works such as 21.1MJ.kg^{-1} (Santos et al. 2019) and 19.2MJ.kg^{-1} (Silva and Mattos, 2020).

The açaí seed samples showed a lower ash content, 0.8% (seed with fiber) and 1.5% (seed without fiber), in compared to coal sample, 15.4%, a desirable value given to the problems intrinsic to biomass ashes such as corrosion, slag and fouling formation in combustion processes due to the lower melting temperatures of some ash components (Teixeira et al., 2012).

4.2 Thermogravimetric Analysis (TGA)

Table 2 shows the temperature data for combustion stage obtained by TGA. Coal sample showed one combustion stage (one DTG peak) in a temperature range of 651-776K. Açaí seed samples showed two combustion stage (two DTG peak) in a temperature range of 524-750K (seed with fiber) and in a range of 540-763K (seed without fiber).

Table 2. Regions and DTG Peak Temperature (K).

Heating Rate (K.min^{-1})	Bituminous Coal			Seed with fiber			Seed without fiber		
	Region	1st Peak	2nd Peak	Region	1st Peak	2nd Peak	Region	1st Peak	2nd Peak
10	651-774	-	734	524-730	554	690	540-746	562	718
20	721-741	-	732	550-747	564	708	554-757	562	729
30	692-776	-	764	548-750	567	676	547-763	569	728

Figure 4 represents the TG and DTG graph for coal sample where we can observe the presence of one combustion stage (second peak). The mass loss is characteristic of char oxidation due to the higher fixed carbon content (49.7%). No significant mass loss signal was identify below 600K due to the lower volatiles content (34.9%).
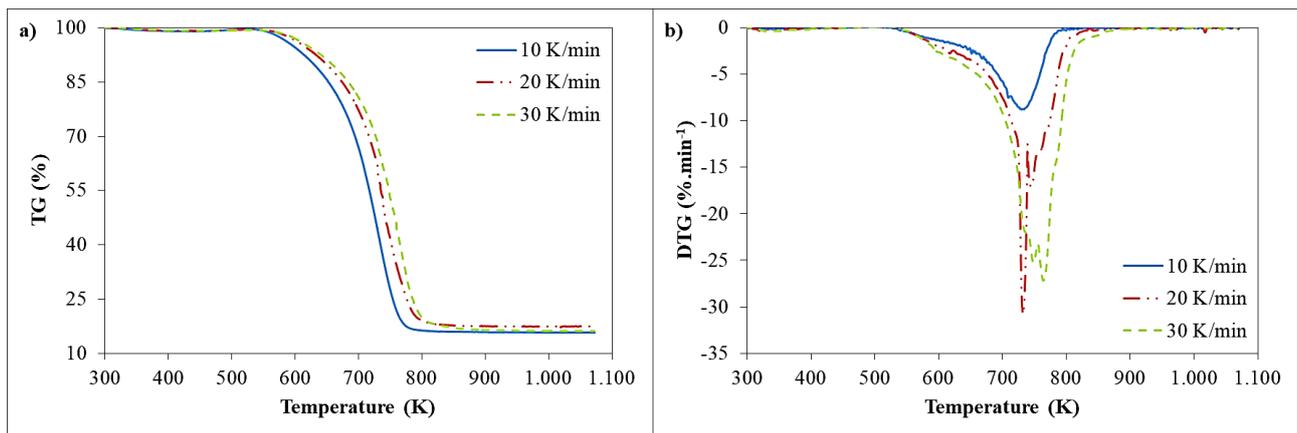

Figure 4. a) TG and b) DTG graph for bituminous coal sample.

The seed with fiber showed two combustion stages, Figure 5. The first stage (volatiles release) was more intense than second stage (char oxidation) due to the higher volatiles content (77.6%). This behavior is common for biomasses that usually have lower ignition temperatures.
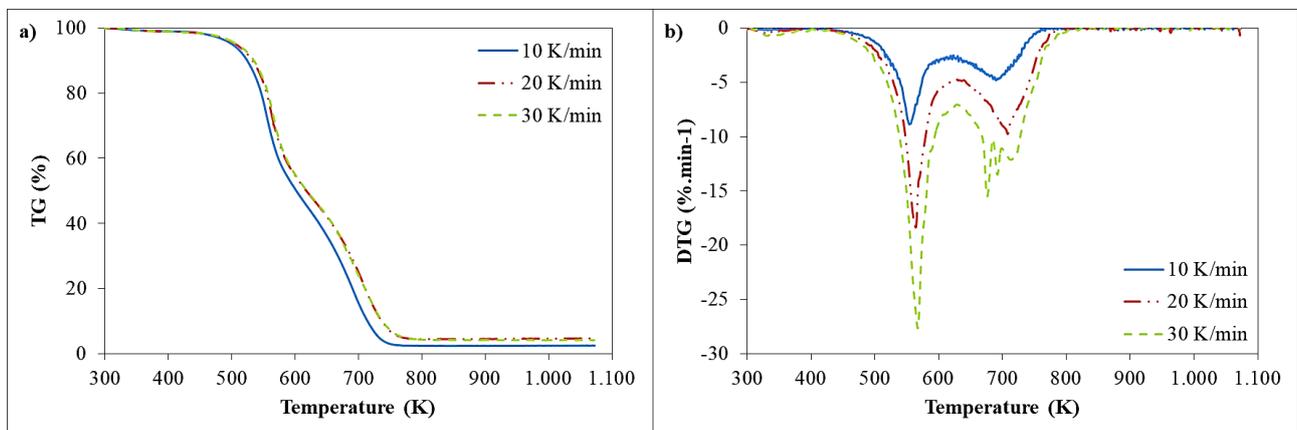

Figure 5. a) TG and b) DTG graph for açaí seed sample with fiber.

The seed without fiber, Figure 6, showed similar behavior to the seed with fiber, with a presence of two combustion stages. The first stage is associated to hemicellulose and cellulose reaction while the second is associated to lignin reaction (Álvarez et al., 2016).
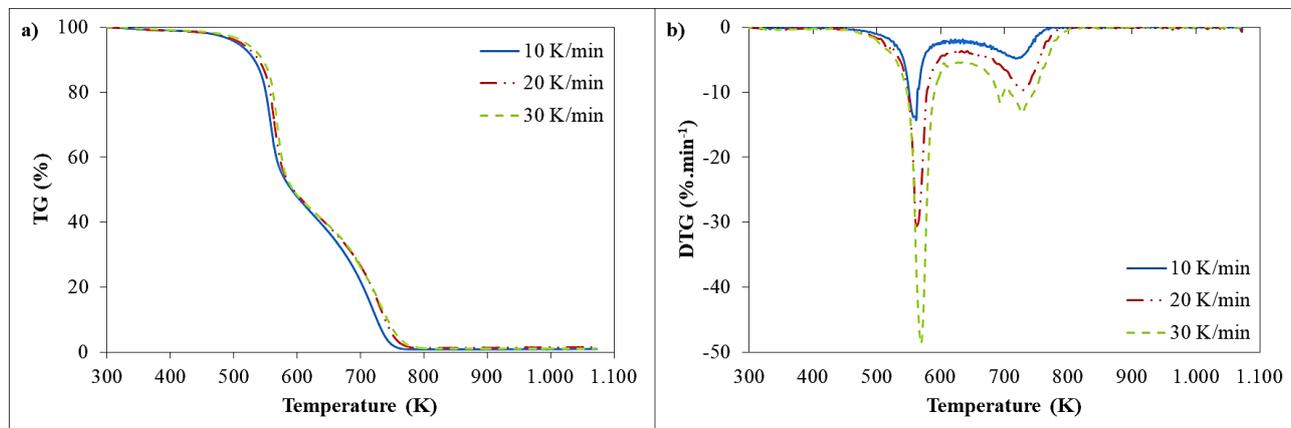

Figure 6. a) TG and b) DTG graph for açaí seed sample without fiber.

Table 2 shows the DTG values (%.min^{-1}) for all samples. For the volatiles release (first peak), the seed without fiber was more intense than the seed with fiber. This could indicate the remove of fiber could make more intense flames in the volatiles release. For char oxidation (second peak), both açaí seed samples showed the same behavior. Coal sample was more intense in the second combustion stage.

Table 2. DTG peak values (%.min^{-1}).

Heating Rate	Bituminous Coal		Seed with fiber		Seed without fiber	
(K.min^{-1})	1st Peak	2nd Peak	1st peak	2nd Peak	1st Peak	2nd Peak
10	-	-8.8	-8.9	-4.8	-14.3	-4.8
20	-	-30.8	-18.4	-9.8	-30.6	-9.7
30	-	-27.2	-27.7	-15.5	-48.8	-13.2

4.3 Kinetic Parameters

The activation energy data was display as a function of conversion degree. The activation energy for coal sample, Figure 7, showed a decrease between 0.05-0.2, an increase between 0.2-0.85 and a decrease between 0.85-0.95. The increase between 0.2-0.85 suggests that more energy is required for the reaction occurs due to the components that react less readily (Deng et al., 2017).
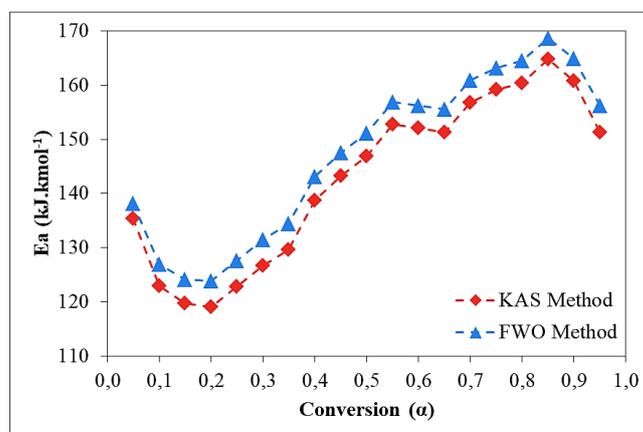

Figure 7. Activation energy for bituminous coal sample.

The correlation coefficient (R^2) showed values in a range of 0.976-0.999 (KAS) and 0.98-0.999 (FWO), a good correlation for coal sample for both isoconversional methods.

The seed with fiber showed a more pronounced variation in activation energy, Figure 8 a), with a decrease between 0.05-0.15, an increase between 0.15-0.35, a decrease between 0.35-0.65 and an increase between 0.65-0.95. This behavior is common for biomass due to the heterogeneous characteristics. Between 0.15-0.65 occurs the volatiles release and accord to Rahib et al., (2020) higher values for activation energy on this process was attributed to the conversion of complex structure of hemicellulose and cellulose.

The decrease between 0.35-0.65 occurs due to the reduction on the volatiles release. The values between 0.65-0.95 are characteristic of char oxidation and showed lower values compared to the first stage, however higher than coal sample. The correlation coefficient (R^2) showed values in a range of 0.781-0.985 (KAS) and 0.8-0.986 (FWO), lower than obtained for coal sample.
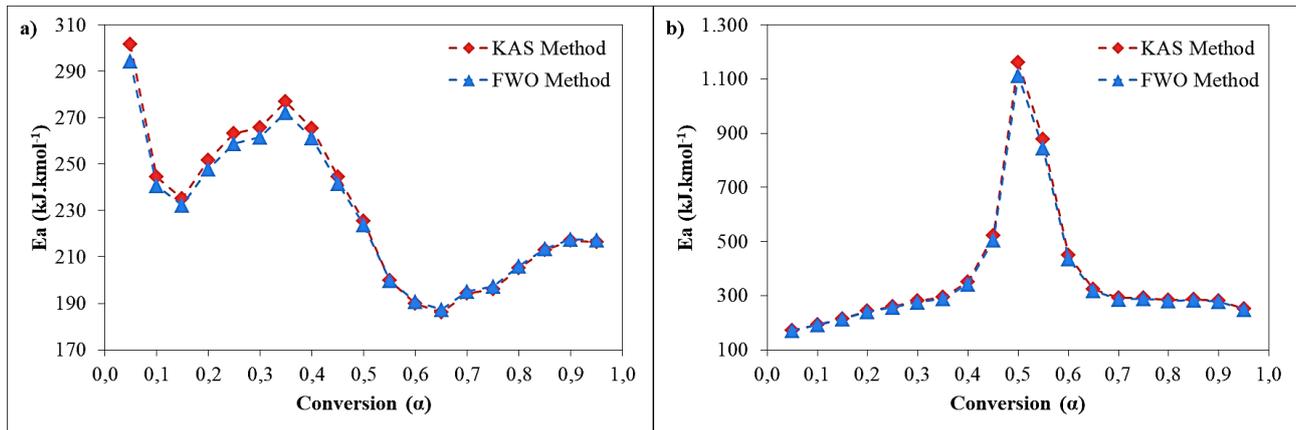

Figure 8. Activation energy for açaí seed with fiber a) and without fiber b).

The seed without fiber showed a different behavior for activation energy, Figure 8 b). It was observed an increase between 0.05-0.4, a steeper increase between 0.4-0.6, and a decrease until 0.95. The behavior observed between 0.4-0.6 was not observed in studies used as a reference in this work, which suggests the need for other kinetic methods for a better evaluation. The correlation coefficient (R^2) showed values in a range of 0.844-1 (KAS) and 0.854-1 (FWO), lower values similar to the seed with fiber.

The lower values for correlation coefficient (R^2) for açaí seed samples, for both isoconversional methods, indicate a great divergence on the results, unlike the values for coal with higher correlation coefficient. The low values may be related to experimental errors or to the type of method used on this work.

Table 3. Activation energy values (kJ.kmol^{-1}).

Conversion Degree (α)	Bituminous Coal		Seed With Fiber		Seed Without Fiber	
	KAS Method	FWO Method	KAS Method	FWO Method	KAS Method	FWO Method
0.05	135.4	138.2	301.5	294.5	170.9	170.6
0.10	122.9	126.9	244.4	240.7	193.1	192.1
0.15	119.7	124.2	235.2	232.2	214.6	212.7
0.20	119.1	123.9	251.6	247.8	243.7	240.4
0.25	122.8	127.6	263.2	259.0	259.9	255.9
0.30	126.7	131.5	265.8	261.5	280.2	275.3
0.35	129.7	134.4	277.0	272.3	294.2	288.7
0.40	138.7	143.2	265.3	261.3	350.9	342.5
0.45	143.2	147.5	244.6	241.8	522.6	505.9
0.50	146.8	151.1	225.4	223.8	1161.8	1113.7
0.55	152.8	156.8	199.9	199.8	878.7	845.0
0.60	152.1	156.2	189.9	190.7	449.1	437.0
0.65	151.3	155.6	186.1	187.3	323.7	318.1
0.70	156.8	160.9	194.1	195.1	290.4	286.8
0.75	159.2	163.2	196.2	197.3	291.1	287.7
0.80	160.4	164.5	205.2	206.1	282.6	279.8
0.85	164.8	168.7	213.0	213.6	286.1	283.3
0.90	160.7	165.0	217.1	217.7	279.6	277.4
0.95	151.3	156.2	216.5	217.3	250.3	249.7

Table 3 shows the activation energy values for all samples. Coal presented lower values of activation energy compared to açaí seed. Coal showed a range of 119.1-164.8 kJ.kmol^{-1} (KAS) and 123.9-168.7 kJ.kmol^{-1} (FWO). Seed with fiber showed 186.1-301.5 kJ.kmol^{-1} (KAS) and 187.3-294.5 kJ.kmol^{-1} (FWO). Seed without fiber 170.9-1161.8 kJ.kmol^{-1} (KAS) and 170.6-1113.7 kJ.kmol^{-1} (FWO). According to Senneca and Cerciello et al., (2023) the values found in the literature present a great variation and in general vary in the range of 10-250kJ.kmol^{-1}.

It was observed that the results of the KAS and FWO methods presented the same behavior for all samples, with a minimal difference, which was expect given the fact that they are different numerical approximations. Rahib et al., (2020) and Rico et al., (2022) obtained the same results in relation to the comparison between the two methods. The seed without fiber showed the highest range for activation energy.

5. CONCLUSIONS

The TGA data allowed the identification of two combustion stages for açaí seed samples by the presence of two DTG peaks. The first stage is related to the volatiles release and the second stage for char oxidation, characteristic for biomass in combustion process. The coal sample presented only one stage for char oxidation, due to higher fixed carbon content and low volatile content, which makes the first stage indistinguishable in the TG and DTG curves.

The first stage for açaí seed was more intense due to the high volatile content, a characteristic behavior for biomasses fuels. The seed without fiber showed higher reactivity in the volatile release than the seed with fiber, which may be associated with the treatment for fiber removal that significantly affects the intensity of volatile release. Regarding char oxidation, both materials of açaí seed presented the same behavior. The intensity of volatile release is the main characteristic of acai seed, which suggests the formation of more intense flames and higher temperatures in the initial combustion stage in comparison with coal.

KAS and FWO methods allowed obtaining the activation energy data in a simply and efficiently, which allowed an understanding the combustion behavior of samples. Both isoconversional methods showed the same behavior for each sample. The activation energy data allowed us to understand the complexity of the reactivity of the açaí seed, in which the variation as a function of the degree of conversion indicates that the material reacts by more than one reaction mechanism and in a complex manner, as expected for biomass fuels. For seed with fiber the values is in concern with literature, however the seed without fiber showed a higher ranger for activation energy values, which need other methods for better understanding e for obtained better correlation coefficient (R^2).

For future works other methods will be used for estimation of activation energy and pre-exponential factor in order to characterize the açaí seed with better precision and for characterize only the fibers of the açaí seed to understanding how the fiber affect the combustion behavior.

6. ACKNOWLEDGEMENTS

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