Journal of Cleaner Production 429 (2023) 139527

Contents lists available at ScienceDirect

Journal of Cleaner Production

journal homepage: www.elsevier.com/locate/jclepro

Irrigation in *Jatropha curcas* L. cultivation and its effect on biomass for bioenergy generation

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A R T I C L E I N F O

Handling editor: Maria Teresa Moreira

Keywords:
Irrigation
Waste management
Physic nut
Energy density
Chemical composition
Biomass pretreatment

A B S T R A C T

The use of biomass as a source of bioenergy has intensified in recent years and has gained strength in replacing fossil fuels and their derivatives. *Jatropha curcas* L. oil is currently used as a raw material for the production of biodiesel through the physical extraction of the fruit, generating a large amount of waste. The heterogeneity characteristics of these materials impose the need for thermal pre-treatments for their recovery, such as torrefaction. Thus, this study aimed to investigate the biomass of *Jatropha curcas* as affected by water availability and its response to the torrefaction process. This research analyzed the biomass physical, chemical, and energy characteristics. TGA/DTG evaluated the thermo-degradation profile, and infrared spectroscopy (FTIR) determined the aromatic chemical structure. The fresh biomass of epicarp and cake presented different behaviors regarding water availability conditions, as variations in lignin contents from 29% to 2.7% and 30.9%–5.7%, respectively, and extractive range from 45.3% to 19.8% and 44.6%–21.6%, respectively. Torrefaction contributed to the increase in physical-chemical characteristics such as lignin and fixed carbon levels, from 29% to 82.4% and 19.8%–52.7%, respectively, and net calorific value of biomass, valuing them for energy use, as well as to decrease in the content of volatile materials from 80.7% to 71.8%. Using renewable biomass of *Jatropha curcas* cake and epicarp for energy purposes contributes to the reduction of environmental impacts by reducing the disposal of these residues in the environment, providing a sustainable and more efficient destination.

1. Introduction

The search for alternative energy sources has intensified in recent years due to the growing need to use renewable fuels, which have remarkable energy efficiency and less environmental impact than fossil fuels (Stolarski et al., 2020; Welfle et al., 2020). Thus, the characterization of biomass is an important issue to be considered in the gradual process of replacing fossil energy sources and their derivatives with sustainable sources. The possibilities for obtaining energy through the utilization of biomass are numerous and must be used efficiently so that they contribute the increase of renewable energy in global energy matrices. In the European Union, for example, energy production from

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https://doi.org/10.1016/j.jclepro.2023.139527
Received 25 May 2023; Received in revised form 8 August 2023; Accepted 27 October 2023
Available online 5 November 2023
0959-6526/

renewable energy sources went from 8.5% in 2004 to 17.5% in 2017, through national action plans (EUROSTAT, 2021).

The greater availability of plantation areas for oilseed plants puts Brazil in a competitive position concerning the potential for producing raw materials for biodiesel compared to most countries in the world (Embaye et al., 2018; Suarez et al., 2009). In Brazil, among the more than 200 species of oilseed species, are the primary raw materials for the production of biodiesel, particularly soybeans (*Glycine* max (L.) Merr.), castor beans (*Ricinus communis* L.), physic nut (*Jatropha curcas* L.), as well as others (Cavalcante Filho et al., 2019).

Jatropha curcas has been used for the production of biodiesel due to the high oil content in its seeds, which varies from 32 to 50% depending on the extraction process, and other desirable characteristics such as high grain yields, good oil quality, and adaptability to different regions (Achten et al., 2010; Santos et al., 2017). The quality of *J. curcas* oil meets international standards, which puts this species in evidence of the capacity to produce biofuels, along with significant government investment and the creation of community projects worldwide. An example of this, the "*Jatropha system*" project, resulted in numerous initiatives to plant the species in the semi-arid tropics (Achten et al., 2010; GEXSI LLP, 2008). These initiatives are due to the species *J. curcas* presenting a production of abundant quantities in the driest regions of Brazil, around 20,000 kg.ha-1, of which 6000 kg.ha-1 is formed by fruits, a product of greater economic interest due to the versatility of uses (Drumond et al., 2016). These actions are also repeated in other regions of the world, such as Zimbabwe and Malaysia, which encourage the planting of these species due to the high production of biomass that can meet the region's high energy demands with its use of bio-oil (Karavina et al., 2011; Riayatsyah et al., 2022).

However, the remarkable adaptability of *J. curcas* to different edaphoclimatic conditions has generated debates regarding the dependence of irrigation for these species (Santos et al., 2018). This shows the need for studies in various regions to evaluate the response of the plant under different irrigation management and mainly the influence of water availability on its chemical composition, such as lignin, holocellulose, and extractives contents (Abou Kheira and Atta, 2009; Achten et al., 2010). Studies carried out in Central America have shown that the species developed better in tropical savanna climates and required a minimum precipitation rate of 944 mm year^{-1} (Achten et al., 2010; Maes et al., 2009), however, it was resistant even in regions with low rainfall of 500 mm year^{-1} with infertile soils and little management (Alherbawi et al., 2021).

When producing biodiesel from *J. curcas*, the removal of the seeds generates a large amount of waste, mainly the epicarp (peels) and the cake (product manufacturing from the pressing of *J. curcas* seeds for the extraction of crude oil). These residues can be used to generate thermal energy through combustion. According to Vale et al. (2011), the epicarp and the cake of *J. curcas* can reach a useable heating value of around 10.92 J/kg and 19.29 J/kg, respectively. These materials must be homogenized before energy valorization using fast, efficient, low-cost treatments due to their physical, chemical and energy attribute when fresh, heterogeneity, high moisture, higher content of volatile materials, lignin, and fixed carbon content, and lower net calorific value. The torrefaction process is an example of a fast and low-cost thermal treatment, which alters the technological properties of biomass and energetically enhances the processing residues of *J. curcas* (C. Chen et al., 2021a; Talero et al., 2019). In contrast to alternative procedures involving thermally modified outputs such as pyrolysis and gasification, there exists no requisite for attaining temperatures exceeding 300 °C (Mendoza Martinez et al., 2021a), thus mitigating wasteful energy dissipation inherent in the process. Furthermore, the torrefaction process gains ascendancy when juxtaposed against hydrothermal carbonization (Mendoza Martinez et al., 2021a, 2021b), given its obviation of the need for sophisticated and high-cost apparatus, thereby driving down the overall cost and rendering it accessible. The new product, known as "torrefied" biomass obtained at temperatures between 200 and 300 °C, has several advantages over fresh biomass, such as higher lignin and fixed carbon contents and lower moisture and volatile material contents (Chen et al., 2022; W.-H. Chen et al., 2021b; da Silva et al., 2018).

Information on the influence of irrigation and torrefaction on the physical, chemical, and energetic composition of epicarp and cake *J. curcas* still needs to be made available. While it is necessary to investigate energy alternatives that contribute to replacing fossil energy sources and their derivatives with sustainable sources, the characterization of alternative materials, such as biomass, becomes relevant globally. Thus, in this study, residual biomasses, epicarp, and cake were investigated from the processing of *Jatropha curcas* grown in two water availability systems and their responses to torrefaction for energy generation.

2. Material and methods

2.1. Experimental area and production of Jatropha curcas biomass

The experiment used biomass from the epicarp and cake of seeds of *J. curcas* cultivated in two areas, one irrigated by central pivot and the other under rainfed conditions. The plants were arranged at a 4 × 3 m spacing (12 m^2 per plant) between rows and plants (Santos, 2016), respectively, totaling 833 plants ha^{-1}. In the irrigated area, management was carried out based on the evapotranspiration of the crop determined by two circular weighing lysimeters located in the center of each area, calibrated and tested by (Flumignan, 2012). Weighing lysimeters readings allows the calculation of the amount of water needed for replacement according to the regular consumption of the plant. Fertilization to meet the crop nutritional demand was carried out in four applications throughout each production cycle, using urea, single superphosphate, and potassium chloride as sources of N, P, and K (12-6-12), respectively, with 200 g of the formulation distributed per plant.

The fruits were harvested when they reached the physiological maturity stage, determined by the yellow color (Jonas et al., 2020). The harvested fruits were placed to dry under the shade at room temperature, being weighed until they reached a moisture content of around 8% for processing. After processing, the epicarp and the cake from the oil seeds extracting process were obtained.

2.2. Thermal torrefaction process of Jatropha curcas biomass

Samples of the epicarp and cake of *J. curcas* were initially dried in an oven at 103 °C ± 3 °C until constant mass. Then, the material was placed inside a hermetically sealed metal torrefaction reactor (Fig. S1, Supplementary Material), equipped with an access door and holes for the insertion of thermocouples to control the temperature and removal of process gases, and then inserted into a muffle furnace, Magnu's 200G analog model. The process temperature started at about 30 °C, with a heating rate of 1.5 °C min^{-1} until reaching the final temperature of 280 °C ± 5 °C (Andrade et al., 2017; Dhungana et al., 2012), and maintained for 60 min. The final 60-min threshold and the heating rate were defined based on preliminary tests carried out with support from the research group Bioenergy and Forest-Based Bioproducts.

2.3. Assessments of fresh and torrefied biomass

The biomass moisture was determined according to the procedures contained in the D1762-84 (American Society for Testing and Materials, 2021). The bulk density of biomass conditions was measured according to the E873-82 standard (American Society for Testing and Materials, 2019). The torrified material yield was obtained by weighing the material before and after each torrefaction process, according to Eq. (1).

$$GY = 100\left(\frac{MF}{MI}\right) \quad (1)$$

where: GY = gravimetric yield of the material after being torrified (%); Mf = final mass of the biomass after being torrefied (g); Mi = initial mass of fresh biomass (g).

The determination of lignin content and biomass extractives were made according to the procedures contained in the standards TAPPI T-222 (TECHNICAL ASSOCIATION OF THE PULP AND PAPER INDUSTRY, 1974) and TAPPI T-12 (TECHNICAL ASSOCIATION OF THE PULP AND PAPER INDUSTRY, 1975). The holocellulose content was obtained by the difference between the initial mass of the extractive-free sample and the total lignin content. The higher heating value (HHV) of the biomass was determined in an adiabatic calorimeter, IKA C-200 (Labcontrol, São Paulo, Brazil), following the EN 18125 standard (Deutsches Institut Für Normung, 2017). The hydrogen content considered for the analysis was the average value found in biomass, about 6% (Huang and Lo, 2020; Wang et al., 2020). The lower heating value (LHV) was determined according to the DIN EN 14918 [27]. Eq. (2).

$$LHV = HHV - 206H \quad (2)$$

where: LHV = lower heating value (kJ kg^{-1}); HHV = higher heating value (kJ kg^{-1}); H = hydrogen content (%).

The determination of the net heating value (NHV) considered the moisture of the materials (Eq. (3)), and the energy density was calculated from the product between the net heating value (NHV) and the bulk density. The Energy Densification Ratio (EDR) was calculated by dividing the higher heating value from fresh biomass and the higher heating value from torrefied biomass, according to the methodology used by (Aguado et al., 2020).

$$NHV = [(LHV - 206H)(1 - 0.01Mwb)] - (23.05Mwb) \quad (3)$$

where: NHV = net heating value at constant volume (kJ kg^{-1}); H = hydrogen content, dry basis %; LHV = lower heating value (kJ kg^{-1}); Mwb = moisture, wet basis (%). The energy of vaporization (constant volume) for water at 25 °C is 41.53 kJ mol^{-1}. This value corresponds to 206 kJ kg^{-1} for 1% (m/m) of hydrogen in the biomass or 23.05 kJ kg^{-1} for 1% (m/m) of moisture, respectively.

The proximate analysis of biomass, contents of volatile materials (VM), ash (AS) and fixed carbon (FC) was carried out in accordance to D1762-84 (American Society for Testing and Materials, 2021), in which the fixed carbon content was obtained by Eq. (4):

$$FC = 100 - (VM + AS) \quad (4)$$

where: FC = fixed carbon content (%); VM = volatile material content (%); AS = ash content (%).

The differential thermogravimetric analysis (TGA) was performed in a TGA-60 Shimadzu equipment, under a nitrogen gas atmosphere, at a constant flow rate of 50 mL min^{-1} and heating rate of 10 °C min^{-1}. The mass used was ±4 mg of material on a dry basis with particle size between 200 mesh and 270 mesh. Thermograms were obtained from room temperature (25 °C) to a maximum temperature of 850 °C. The carbonaceous aromatic chemical structure was evaluated with the aid of a Bruker Fourier Transform Infrared (FTIR) spectrometer, Tensor-27 model, in the experimental mode of attenuated total reflection (ATR) with 32 scans and spectral amplitude between 600 and 4000 cm^{-1} with sample in an ATR accessory with ZnSe crystal. To analyze the microstructures of the fresh and torrefied biomass produced, samples from the transversal plane were analyzed by Confocal microscopy using the Olympus OLX 4000 microscope. The samples were not subjected to specific treatment since the original surface of the sample was analyzed.

The preparation of the samples and the determination of the Polycyclic Aromatic Hydrocarbons (PAHs) in the biomass were carried out in accordance to the Environmental Protection Agency EPA 3550C (Environmental Protection Agency, 2007a) and EPA 8270D (Environmental Protection Agency, 2007b) using a gas chromatography technique coupled to a mass spectrometer (GC/MS). PAHs investigated were: Acenaphthene, Acenaphthylene, Anthracene, Benzo [a]anthracene, Benzo(a)pyrene, Benzo [b]fluoranthene, Benzo [g,h,i]perylene, Benzo [k]fluoranthene, Chrysene, Dibenz [a,h]anthracene, Phenanthrene, Fluoranthene, Fluorene, Indeno [1,2,3-cd]pyrene, Naphthalene and Pyreno.

2.4. Data analysis

The data obtained were evaluated by the Kolmogorov-Smirnov test to verify normality. In addition, the Levene test was performed to test the homogeneity of the variances. The analysis of variance was performed following a split-plot utterly randomized design (dry and irrigated regime x fresh and torrefied biomass) and five replications. For the multiple comparison test of means, the Tukey test was used. The tests were done at a 95% confidence interval. For principal component analysis (PCA) (Hair et al., 2009), physical properties (moisture and bulk density), chemical (lignin, holocellulose, and extractives), proximate analysis (VM, AS, and FC), and energy (HHV, LHV, NHV, and energy density) parameters of the biomass were included. The scores of the dispersion of the main components allowed the grouping of biomass with similar characteristics aiming at the production of bioenergy. The analyses were performed with the support of the R Core Team (R Core Team, 2022).

3. Results and discussion

3.1. Physical and chemical properties of Jatropha curcas biomass

Fig. 1 presents the moisture of the epicarp and biomass cake, fresh and torrefied, of *J. curcas* according to the water regime. See Supplementary Material Table S1 for detailed information on means, dispersion, and statistical analysis.

In general, the *J. curcas* epicarp showed greater moisture when compared to the cake. Vale et al. (2011) associate the lower moisture content of the cake with the effect of pressing the seed in the oil extraction process. Nevertheless, the epicarp of seeds produced by irrigated plants showed higher moisture content than plants grown under rainfed conditions. In this study, the torrefaction influenced biomass hygroscopicity, making it less moist (Pimchuai et al., 2010). According to Ciolkosz and Wallace (2011), the reduction in water content occurs due to the decomposition of hemicellulose, cellulose, and lignin, making them more susceptible to the release of water molecules stored at the cellular level and the decrease in hydrogen bonds due to the removal of OH groups.

The bulk density of the cake (Fig. 1), in general, was larger than the density of the epicarp. However, there was a significant difference between the density of fresh and torrefied cake obtained from irrigated plants compared to the cake from the rainfed regime (Table S1, Supplementary Material). Irrigation did not promote any significant difference in the fresh biomass of epicarp compared to the rainfed treatment. Vale et al. (2011) obtained similar results of the cake bulk density when compared to that of the epicarp, inferring that the smaller particle size of the cake, due to its handling and the pressing process, both for the extraction of the oil, contributed to the increase in density. This same result was observed by Tomeleri et al. (2017), where the density of the epicarp is similar to the value found in the present study and is considerably lower than the density attributed to the pie of *J. curcas*. After torrefaction, all materials showed significant differences compared to the wet condition. da Silva Grassmann et al. (2016) observed similar results when applying the torrefaction process to wood residues, which showed a significant decrease in bulk density due to the degradation of their constituents by the action of heat. In addition, the fresh cake, which has a higher density than the epicarp, suffered a more

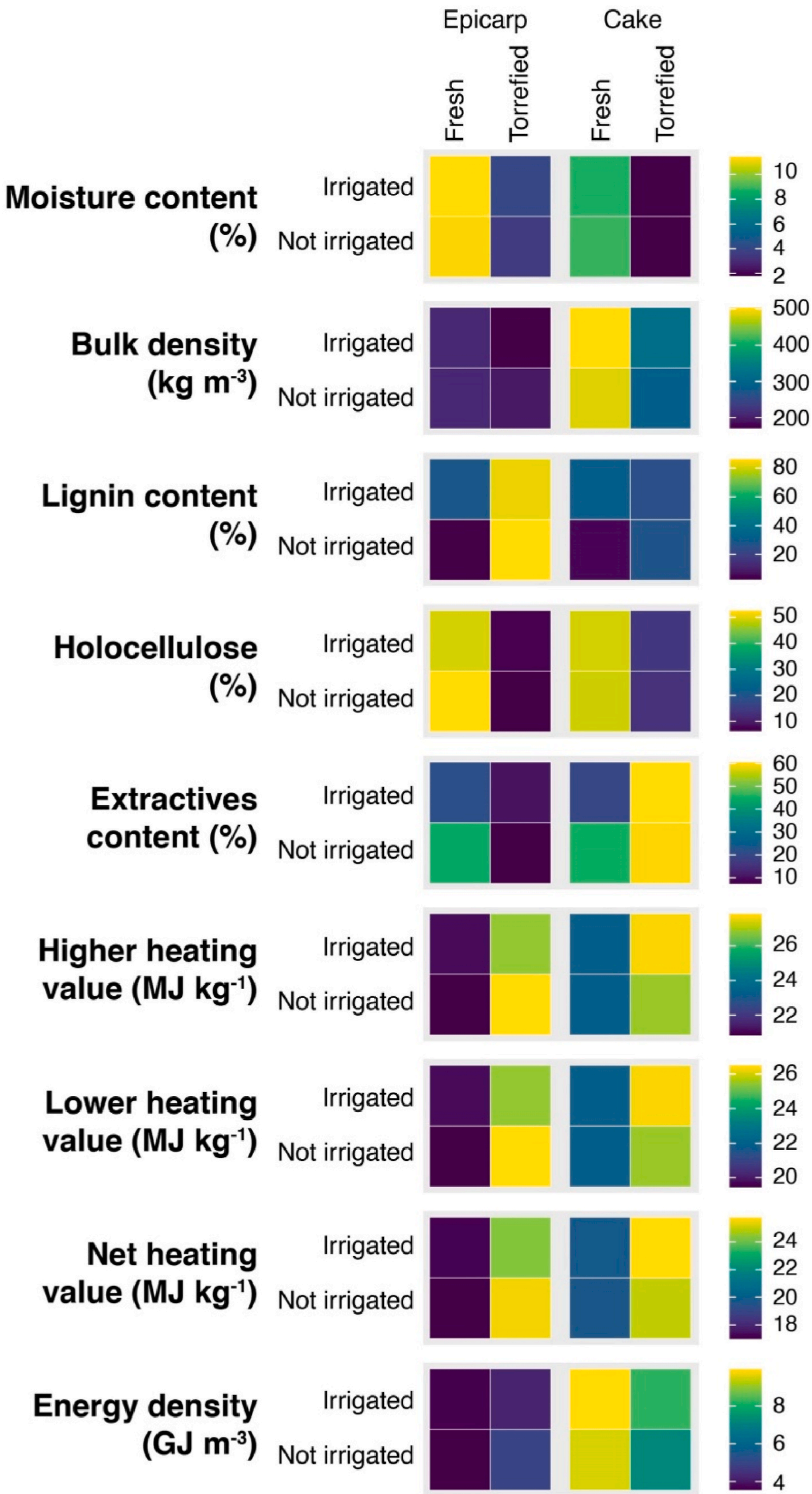

Fig. 1. Physical, chemical and energy variables investigated in *J. curcas*.

significant reduction in this variable after torrefaction. The biomass of epicarp showed an average 15% decrease in its bulk density, whereas the cake showed an average loss of 38%, a result similar to that found by da Silva Grassmann et al. (2016), who observed that wood wastes with lower density showed less reduction in mass per unit volume after the torrefaction process.

The torrefaction yield was approximately 52 and 75% for the epicarp and cake of *J. curcas*, respectively, with no significant difference between the biomass of the irrigated plants and that of plants that did not receive irrigation. This difference may be associated with the chemical constituents of biomass (Fig. 1) which are responsible for the final product after torrefaction (W.-H. Chen et al., 2021b; da Silva et al., 2018). It was possible to notice the color change of the biomass after torrefaction (Fig. S2, Supplementary Material). Note that the fresh epicarp had structures on its surface with a different aspect from those observed in the torrefied epicarp, showing the degradation by the action of temperature in the torrefied biomass. In the fresh cake, no structures were observed on its surface, unlike the torrefied cake, where small white dots were observed, suggesting the possible presence of mineral ash in the material. Fig. 1 shows the results obtained from the chemical analysis of the levels of lignin, holocellulose, and extractives, respectively, of the fresh and torrefied biomass of *J. curcas.*

The lignin content in the fresh biomass samples of epicarp and cake from irrigated plants had higher values in comparison to the biomass of plants grown without irrigation (Fig. 1). Singh et al. (2013) and Santos (2016) observed that the fruits of *J. curcas* from plants with irrigated treatment showed better aspects than those of non-irrigated treatment, showing better growth, productivity, and seed's yield. After the biomass torrefaction process, the lignin content in the epicarp samples was higher than that of the cake since lignin is responsible for forming a solid product (W.-H. Chen et al., 2021b; Mamvura et al., 2018), due to its complex composition, becoming the main constituent after the torrefaction process. Thus, there is an increase in lignin content after torrefaction, and positive correlations with higher calorific value, fixed carbon content, and gravimetric yield are obtained with increased lignin content (Benites et al., 2018).

The holocellulose content of the biomass showed no difference as a function of the water regime, except for the epicarp of non-irrigated plants, which showed a higher value than the other residues. Benites et al. (2018), studying the effect of irrigation on eucalyptus clones, did not observe an increase in holocellulose content with the use of irrigation. The holocellulose content of the biomass decreased significantly with the torrefaction process, especially in the epicarps, since the final temperature of the process close to 300 °C contributed mainly to the degradation of the hemicellulose, which has the lowest degradation range (200–300 °C) due to its branched and amorphous structure (Chen et al., 2018; Mamvura et al., 2018). The decrease in the holocellulose content is advantageous for energy purposes because cellulose and hemicelluloses have negative correlations with gravimetric yield, as they are responsible for the release of gases and liquid products during torrefaction (Benites et al., 2018; Chen et al., 2018).

The extractives content was higher in the fresh biomass from non-irrigated plants, showing that the water condition influenced the extractives present this fact was also observed by Pedro de Sousa Andrade et al. (2017). The high content of extractives found in the biomass of *J. curcas* can be related to the high content of oil contained in the seeds. Some studies show that the oil content in *J. curcas* seeds responds positively to irrigation (Abou Kheira and Atta, 2009; Kaushik and Bhardwaj, 2013) however Pedro de Sousa Andrade et al. (2017) showed that drought conditions could contribute to the improvement of the chemical composition of *J. curcas* oil, making it more efficient for the production of biodiesel by increase the oleic acid content. The extractive content found in the biomass after torrefaction was higher in the cakes than in the epicarps, which may be an indication of the presence of residual oil in the cakes even after the torrefaction process, which was not enough to cause the total thermo-degradation of the oil present in the biomass. Silva et al. (2016), studying the thermo-degradation in soy biodiesel, observed that temperatures above 280 °C are necessary to start the biodiesel thermo-degradation.

Fig. 1 presents the higher, lower and net heating values, respectively, of the fresh and torrefied biomass of *J. curcas*. Variations were observed only between the biomass types, with the cakes showing higher heating value compared to the epicarps (Fig. 1). These differences may be related to the higher ash content of the epicarps, which culminates in a decrease in heating value (Benites et al., 2018). The greater net heating value of the cakes may be related to the presence of residual oil in the biomass, results that were also observed by Vale et al. (2011). For the torrefied biomass, there was a significant increase in the calorific value in both materials, since the torrefaction process contributes to the increase in the fixed carbon content and, consequently, causes an increase in the calorific value (Benites et al., 2018; Gan et al., 2019; Yang et al., 2020). The results obtained for the energy density (ED) of the fresh and torrefied biomass of *J. curcas* are presented in Fig. 1.

The energy density of the *J. curcas* biomass showed a behavior similar to the bulk density, so the higher values of bulk density contributed to the increase in the energy density of the biomass (Pedro de Sousa Andrade et al., 2017; Chen et al., 2015). The slight variation in the net heating value did not contribute to the increase in energy density, mainly in the torrefied biomass. This fact is due to the directly proportional relationship between these variables, bulk density, energy density, and net heating value, also observed by Souza and Vale (2017). The cakes showed higher energy density values compared to the epicarps (fresh and torrefied); however, the energy density of the torrefied cakes was lower than the fresh cakes due to the loss of the density of this biomass in the roasting due to its greater bulk density before the process (da Silva et al., 2016). As for the water conditions under study, the cake from plants grown under irrigation showed a higher energy density. The increase in the fixed carbon content in the torrefaction process resulted in an increase in the Energy Densification Ratio (EDR) of *J. curcas* biomasses (Table S2, Supplementary Material) and contributed to the increase in the energy density of the epicarp. However, it was observed that torrefaction promoted a decrease in energy density (ED) for cakes. This decrease can be attributed to the lower ED of the cake compared to the epicarps after the torrefaction process, negatively influencing the energy density in the torrefied biomass.

The values obtained for the contents of volatile materials, ash and fixed carbon are shown in Fig. 2 (Table S2, Supplementary Material) for fresh biomass and for torrefied biomass.

As for the content of volatile materials (VM), significant differences were observed in the cake, fresh and torrefied, and in the torrefied epicarp, as a function of the water regime, with higher values of these biomasses when coming from irrigated plants. Such differences can be attributed to the fact that the water that constitutes the biomass comprises part of the gases that are condensable at room temperature, which are classified as volatile materials when present in the biomass, contributing to the increase of this content (Macedo et al., 2014; Neves et al., 2011). However, the fresh epicarp did not show any significant difference regardless of the presence or not of irrigation. In general, the torrefied biomass showed lower values of volatile materials compared to fresh biomass due to the action of temperature in the torrefaction process (close to 300 °C), which promotes the release of permanent and condensable gases at room temperature, culminating in the decrease of volatile materials present in the biomass after torrefaction (Chen et al., 2015; Neves et al., 2011).

The fixed carbon content (FC) showed significant differences as a function of the water regime and the torrefaction, fresh or torrefied epicarp had higher FC than the fresh or torrefied cake (Fig. 2). Macedo et al. (2014), observed higher FC in the epicarp of *J. curcas* when compared to other biomass such as sugarcane bagasse (*Saccharum officinarum* L.), elephant grass (*Pennisetum purpureum* Schum var. Mineiro) and bamboo (*Bambusa vulgaris* ex JC Wendl. Var. *vulgaris*). The high FC of the epicarp can be explained by the high content of lignin, which has a

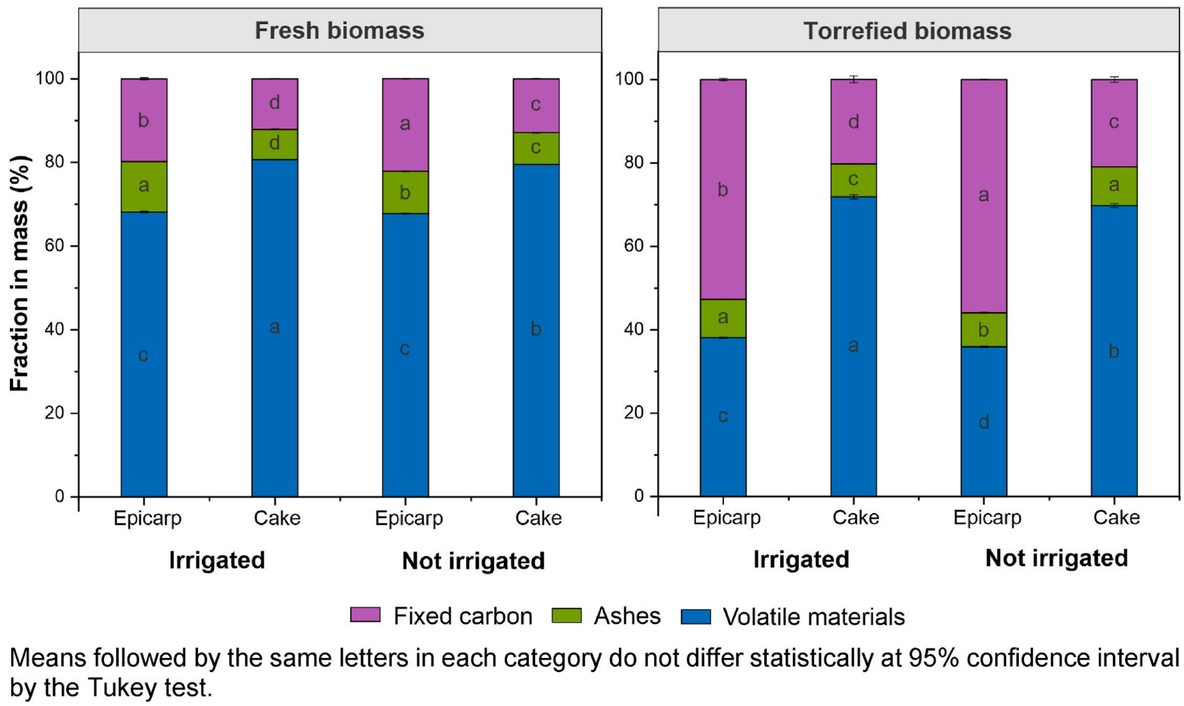

Fig. 2. Proximate composition of the fresh biomass and torrefied biomass of *J. curcas*, grown under different water regimes. Means followed by the same letters in each category do not differ statistically at 95% confidence interval by the Tukey test.

positive correlation since the high percentage of elemental carbon in the lignin structure contributes to the increase in FC (Benites et al., 2018; Yang et al., 2020). The fresh and torrefied biomass from irrigated plants showed lower average FC than those from non-irrigated plants.

The ash content (AS) of irrigated plants' fresh epicarp was higher than that of the non-irrigated plants, unlike the cake, in which the ash content in the cake from non-irrigated plants was higher than that of the cake from plants subjected to irrigation. In general, the epicarps had higher ash contents compared to the cakes, a result similar to that found by Vale et al. (2011) when studying *J. curcas,* being, respectively, 14.4 and 7.9%, also similar to the value found of 7.7% by P. Tomeleri et al. (2017) for the epicarp. Some authors attribute these differences to the presence of inorganic materials in the biomass, such as potassium, calcium, iron, and magnesium, among others, originating in part from the fertilization or origin of the biomass (da Silva et al., 2018; Dai et al., 2019; Gan et al., 2019; Vale et al., 2011). Another fact was that the epicarps ash content decreased after torrefaction, which contributed to the increase of this variable (Álvarez et al., 2018; Dai et al., 2019; Gan et al., 2019), due to the degradation of organic components.

The PCA score analysis showed four biomass groups with different energy properties (Fig. 3).

The two types of fresh biomass were separated into distinct groups (groups 1 and 2), demonstrating that their physicochemical and energy differences had a more substantial effect than the type of irrigation. Overall, this did not significantly affect these properties. Thus, irrigated and non-irrigated biomass are part of the same group. Torrefaction was responsible for the segregation of biomass in groups 3 and 4, in which it was again observed that the type of irrigation did not significantly influence the characteristics of the torrefied biomass. However, the biomass type proved significant for the physicochemical and energy properties after the heat action process.

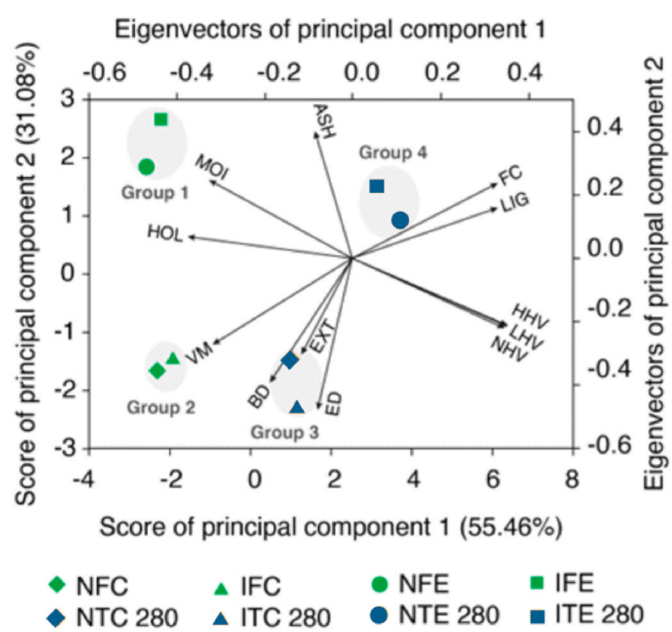

Fig. 3. Principal component analysis (PCA) of epicarp and cake of *J. curcas*, grown under different water conditions. Where: IFE = irrigated fresh epicarp; ITE 280 = irrigated torrefied epicarp; NFE = non-irrigated fresh epicarp; NTE 280 = non-irrigated torrefied epicarp; IFC = irrigated fresh cake; ITC 280 = irrigated torrefied cake; NFC = non-irrigated fresh cake; NTC 280 = non-irrigated torrefied cake.

3.2. *Differential thermogravimetric analysis of Jatropha curcas biomass*

Figs. 4 and 5 show the thermogravimetric curve of the fresh and torrefied epicarp and cake of the biomass of *J. curcas*. The first stage of mass loss occurs from 50 to 150 °C (Fig. 4A) due to the loss of biomass moisture. The thermal decomposition of holocellulose represents the second stage, contributing to the loss of mass that occurred from the temperature of 150 to approximately 270 °C, followed by the third stage, when cellulose degradation occurs at temperatures between 270 and 350 °C (Gan et al., 2019; Sricharoenchaikul and Atong, 2009; Varma and Mondal, 2018). The first stage of mass loss occurs from 50 to 150 °C

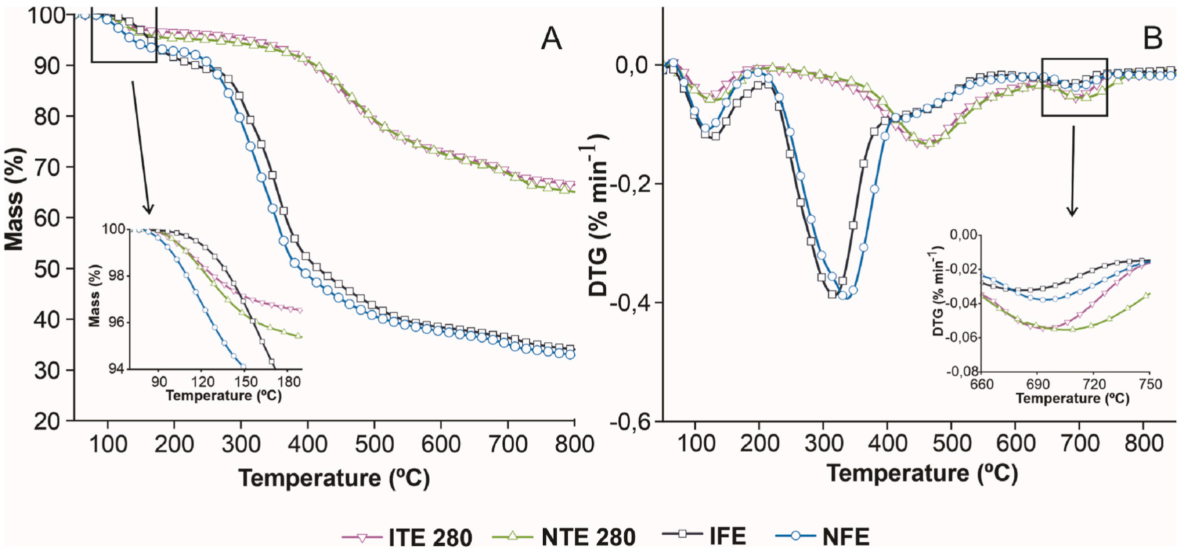

Fig. 4. TGA thermogravimetric analysis (A) and derivative of the DTG thermogravimetric analysis (B) of the fresh and torrefied epicarp of *Jatropha curcas*, grown under different water conditions. Where: IFE = irrigated fresh epicarp; ITE 280 = irrigated torrefied epicarp; NFE = non-irrigated fresh epicarp; NTE 280 = non-irrigated torrefied epicarp.

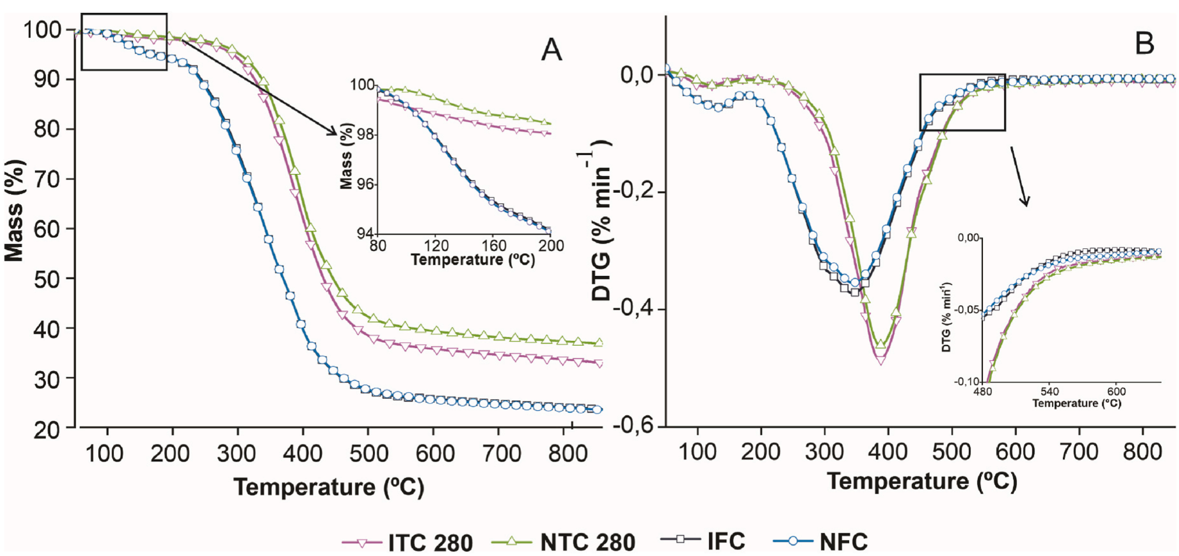

Fig. 5. TGA thermogravimetric analysis (A) and derivative of DTG thermogravimetric analysis (B) of fresh and torrefied cake of *Jatropha curcas*, grown under different water regimes. Where: IFC = irrigated fresh cake; ITC 280 = irrigated torrefied cake; NFC = non-irrigated fresh cake; NTC 280 = non-irrigated torrefied cake.

(Fig. 4A) due to the loss of biomass moisture. The thermal decomposition of holocellulose represents the second stage, contributing to the loss of mass that occurred from the temperature of 150 to approximately 270 °C, followed by the third stage, when cellulose degradation occurs at temperatures between 270 and 350 °C (Gan et al., 2019; Müsellim et al., 2018). Fig. 4B presents the derivative of the curve (DTG) of the TGA, which shows the mass loss reactions that occurred in the biomass of *J. curcas* with the action of temperature. The inflections observed in the DTG curves occurred at the same intervals observed in Fig. 4A, suggesting the presence of mass loss reactions in the intervals from 50 to 150 °C, 150–270 °C, 270–350 °C and at higher temperatures than 400 °C. The most evident loss of mass was observed in the fresh epicarps, given the non-degradation of the chemical components of this material, unlike the torrefied epicarps, which showed less loss of mass with the action of heat due to the lower content of holocellulose and higher content of lignin, making this biomass more thermally resistant. Fig. 5 shows the thermogravimetric analysis of the fresh and torrefied cake by *J. curcas*.

For the *J. curcas* cakes, the stages of mass loss (Fig. 5A) were similar to those observed in the epicarps (Fig. 4A), with the exception of the absence of the torrefied cakes' first stage, which occurs in the range from 50 to 150 °C and relates the loss of mass of the material with the loss of moisture, corroborating the low moisture of the cakes after torrefaction, observed in Fig. 1, so that the degradation of its chemical components also occurred within very similar temperature ranges. However, there is a greater mass loss by the cakes than the epicarps, mainly in the fresh cakes. The stages of mass loss of the biomass mentioned above were evidenced and confirmed by the curve of the TGA derivative (Fig. 5B), which shows the loss reactions and the temperature intervals in which they occurred. However, unlike fresh cakes, the torrefied cakes did not have significant mass loss between 50 and 150 °C (the first stage, which is related to the loss of water from the biomass), highlighting the lower moisture of this material, promoted by the torrefaction (Gan et al., 2019; Pimchuai et al., 2010). However, despite the absence of the first stage (Fig. 5A), the torrefied cakes showed the same tendency of loss of mass as the fresh cakes, being related to the lower lignin content of this material, which contributes to the lower thermal resistance of the cakes despite being torrefied.

3.3. Fourier transform infrared spectroscopy (FTIR)

The FTIR analysis spectra of *J. curcas* biomass are shown in Figs. 6 and 7 for fresh and torrefied biomass.

The fresh and torrefied epicarp showed similar trends, but the spectra of the torrefied epicarps showed that some bands disappeared, which can be attributed to the breakdown of chemical bonds as a result of the temperature. In the irrigated fresh epicarp (IFE) and the non-irrigated fresh epicarp (NFE), the associated hydroxyl functional group (OH bonds), which ranged from 3600 to 3200 cm^{-1}, became more evident (Gan et al., 2019; Pimchuai et al., 2010). The presence of this functional group is related to the constituent water in the material because compared to the fresh epicarp, the irrigated torrefied epicarp (ITE 280), and the non-irrigated torrefied epicarp (NTE 280), this band appears more discreetly in the spectra since torrefaction contributes to the decrease of moisture in the material. The band between 3000 and 2850 cm^{-1} indicated the presence of aliphatic groups (elongation of CH bonds) (Gan et al., 2019; Li et al., 2018),] in NFE and evidently in IFE due to irrigated cultivation, which increases the oil content in the seeds (Abou Kheira and Atta, 2009; Kaushik and Bhardwaj, 2013), suggesting a higher content of aliphatic groups in this material.

The irrigated torrefied epicarp (ITE 280) and non-irrigated torrefied epicarp (NTE 280) showed lower intensity of the aliphatic groups, demonstrating that the torrefaction caused the degradation of these functional groups in this type of biomass. Between 1745 and 1700 cm^{-1}, the presence of C=O stretch (ketones, carboxylic acids, esters, and aldehydes) was observed in the spectra of fresh epicarps (Salema et al., 2014), mainly with greater intensity in irrigated fresh epicarp (IFE), due to the contribution of water availability to the increase of these functional groups. In the irrigated torrefied epicarp (ITE 280) and non-irrigated torrefied epicarp (EST 280), these functional groups were absent due to volatilization caused by the action of temperature. However, nitro-compounds were present at 1550 cm^{-1}, which facilitated the exothermic reaction. The spectra of the cakes and epicarps of *J. curcas* showed differences in the bands due to torrefaction (Fig. 7).

The hydroxyl functional groups present between 3600 and 3200 cm^{-1} were superior in the irrigated fresh cake (IFC) and in non-irrigated fresh cake (NFC), showing that torrefaction reduces the biomass moisture (Gan et al., 2019; Pimchuai et al., 2010) and, consequently, contributes to the increase of the net heating value, since this functional group is related to moisture present in the biomass. Aliphatic groups between 3000 and 2850 cm^{-1} were also observed in the cakes. On the other hand, the cakes, even being torrefied, showed no decrease in this functional group since the temperature of the torrefaction was not sufficient to cause the volatilization of the oil that constitutes the cakes (Kaushik and Bhardwaj, 2013), resulting in the same intensity of the bands of the aliphatic groups for both biomass, fresh and torrefied. The irrigated fresh cake (IFC) and non-irrigated fresh cake (NFC), between 1745 and 1630 cm-1 showed several C=O functional groups such as esters, aldehydes, ketones, and amides that are absent and at low intensities in irrigated torrefied cake (ITC 280) and non-irrigated torrefied cake (NTC 280), due to the degradation of these groups caused by the torrefaction temperature.

The torrefied biomass of *J. curcas*, epicarp and cake, showed levels of PAHs below the limit of qualification (LQ, <0.01 μg kg^{-1}). One of the chromatograms obtained is presented as a supplementary material (Fig. S3) to this research article. Despite being detected in biomass, the values can be considered well below the limits suggested by environmental control agencies in the world on emissions of PAHs (European Parliament and of the Council, 2004). Heat promotes the elimination of volatile materials from biomass, which contributes to the reduction of PAH levels supposedly present in the material (Dias Júnior et al., 2018). Torrefaction, because of its maximum temperature of 300 °C, may not effectively contribute to the total elimination of PAHs when compared to more abrupt processes of heat action, such as pyrolysis, which reaches temperatures above 500 °C. In general, the values of these compounds for biomass are considered satisfactory to enable their use in combustion.

3.4. Practical and policy implications and future perspectives

The Brazilian energy matrix stands out for presenting 40,4% of its production from renewable energy sources, such as biomass, biofuels,

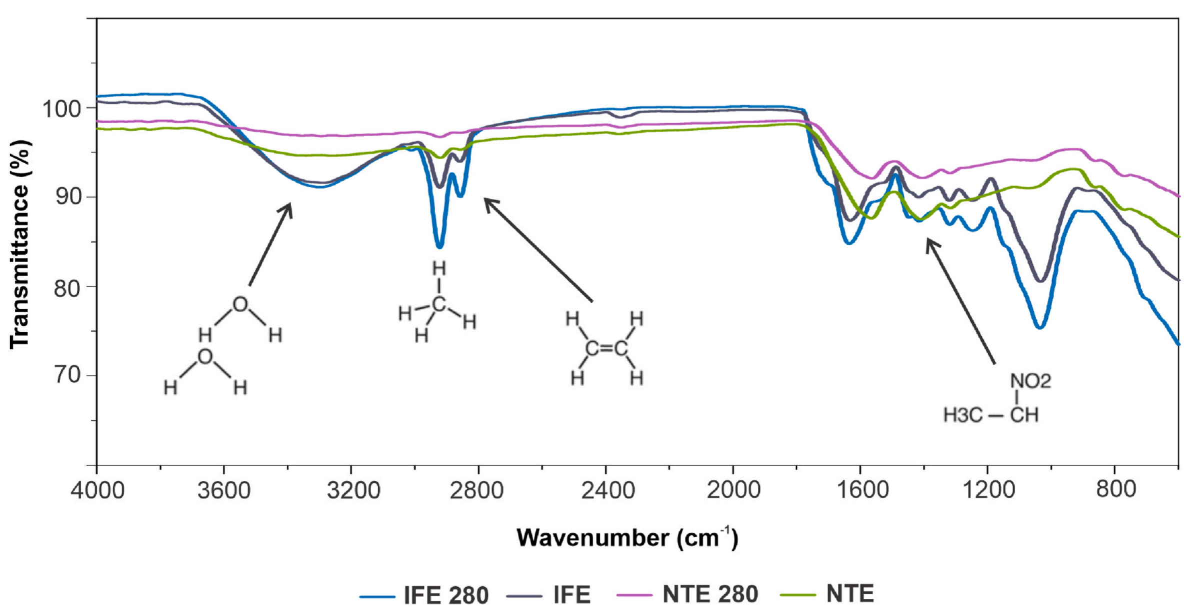

Fig. 6. FTIR spectra of the epicarp of *Jatropha curcas*, where: ITE 280 – irrigated torrefied epicarp, IFE – irrigated fresh epicarp, NTE 280 – non-irrigated torrefied epicarp, and NFE – non-irrigated fresh epicarp.

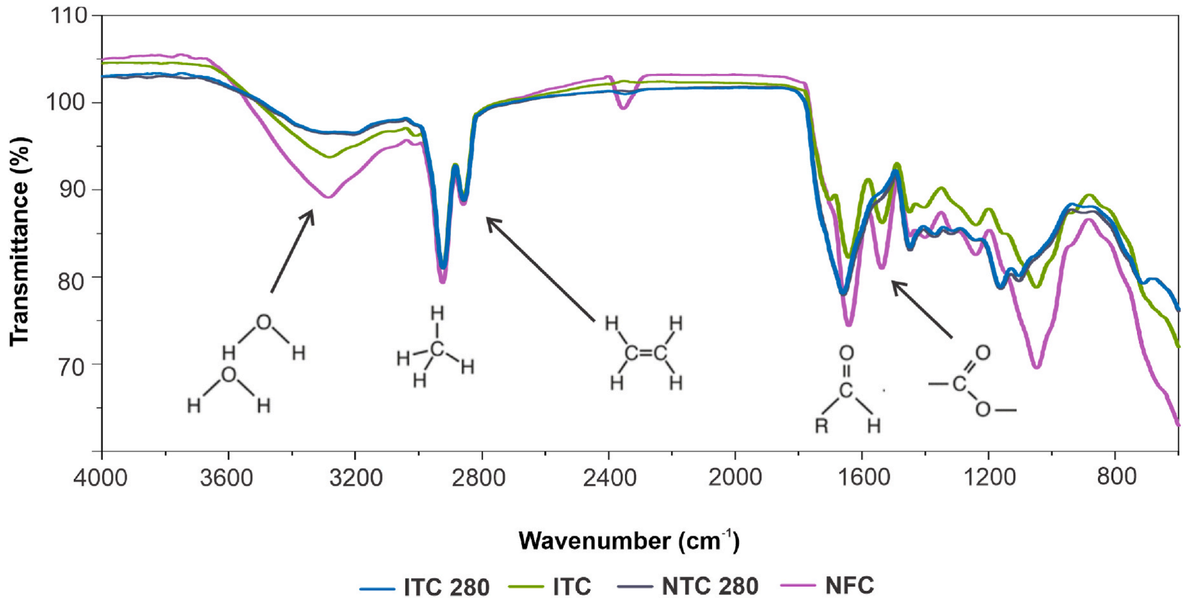

Fig. 7. FTIR spectra of the cake of *Jatropha curcas*, where: ITC 280 – irrigated torrefied cake, IFC – irrigated fresh cake, NTC 280 – non-irrigated torrefied cake, and NFC – non-irrigated fresh cake.

and hydraulic energy (Empresa de Pesquisa Energética, 2020) however, the share of biomass comprises 23.8% of the Brazilian energy matrix. This demonstrates the need to expand the use of residual biomass in energy generation. The Brazilian government estimates that in 2050, 31% of the total biomass used for energy purposes will come from agricultural waste (Ministério de Minas e Energia, 2020). Also, the increase in demand for biodiesel production due to new legislation (Conselho Nacional de Política Energética, 2018) directly contributes to the increase in the generation of wastes from *J. curcas*. In Brazil, the incentive of the National Solid Waste Policy (Brasil, 2010) in energy valorization waste can contribute to increasing the participation of this renewable source and contribute to the objectives of sustainable development (objectives 7 - Affordable and Clean Energy, 12 - Responsible Consumption and Production and 13 - Climate Action, mainly) and ensuring the correct use of these wastes in the context of the circular bioeconomy. Furthermore, these studies with *J. curcas* residues for energy purposes not only contribute to the implementation of a new source of energy for Brazil but also for other countries. Therefore, we recommend that future research be focused on: (1) pelletization studies of the torrefied biomass, including pie blends and epicarp to reduce the ash content of the pellets; (2) analyses of combustibility, kinetic parameters, and gas emissions resulting from the thermal decomposition of residual biomass; (3) characterization of ash and ash-forming elements and the possible impact on the management and maintenance of firing equipment, and (4) quantification of the Brazilian production of biomass residues from the production of biodiesel and feasibility studies of the energy use aiming to contribute to the low carbon economy.

4. Conclusion

The chemical composition of the biomass was susceptible to the water regime, with low lignin content and higher extractives content when not irrigated. The cake of *Jatropha curcas* from irrigated plants stood out for energy use, as it has the highest energy density and net heating value. However, when fresh, this biomass is of heterogeneous composition and can exhibit physical-chemical characteristics that may not be beneficial for its energy use if it is fresh. Despite decreasing the energy density of the biomass, the torrefaction process contributed to the increase of the net heating value and the physical-chemical homogenization of the material. Using biomass from the cake and epicarp of *Jatropha curcas* seeds for energy purposes may be feasible for reducing environmental impacts through the removal of this material from the environment, providing a more sustainable destination for the solid residues generated in the extraction of *Jatropha curcas* oil.

CRediT authorship contribution statement

Luis Filipe Cabral Cezario: Conceptualization, Methodology, Validation, Formal analysis, Investigation, Data curation, Writing – original draft, Visualization, Supervision. **Ananias Francisco Dias Júnior:** Conceptualization, Methodology, Validation, Resources, Writing – review & editing, Supervision, Project administration, Funding acquisition. **Álison Moreira da Silva:** Validation, Formal analysis, Data curation, Writing – review & editing. **Otávio Neto Almeida Santos:** Resources, Validation. **João Gilberto Meza Ucella-Filho:** Data curation, Writing – review & editing. **Thiago de Paula Protásio:** Validation, Writing – review & editing. **Demetrius Profeti:** Validation, Resources, Writing – review & editing. **Daniel Saloni:** Writing – review & editing. **Patrick Rousset:** Writing – review & editing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

Acknowledgments

To the research group Bioenergy and Forest-Based Bioproducts and the Biomass Energy Laboratory of the Federal University of Espírito Santo (LEB/UFES) in Brazil for their support in carrying out the various analyses. This research had the financial support of the Fundação de Amparo à Pesquisa e Inovação do Espírito Santo (FAPES).

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.jclepro.2023.139527.

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