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Preparation of activated carbon from babassu endocarpunder microwave radiation by physical activation

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Abstract. Babassu endocarp were used to prepare activated carbons by physical activation via microwave radiation for the first time. The pyrolysis temperature was 600°C and the derived biochar were activated in CO_2atmosphereat 700, 750 and 800°C for 30 min. The material was characterized using scanning electron microscopy (SEM). The porous properties of the activated carbons obtained including the Brunauer-Emmett-Teller (BET) surface area, pore volume, and average pore diameter were determined by nitrogen adsorption isotherms at 77.32 K. The experimental results showed that most pores occurred during the activation predominantly as micropores. Endocarp babassu can be used as precursor to produce activated carbon with a rather well-developed porosity by pyrolysis and physical activation by two-steps with CO_2 activation via microwaves radiation. The activated carbon, with a low production cost, could be suitable for applications in gaseous pollutant adsorption, adsorb iodine, methylene blue, and residual chlorine.

1. Introduction

Babassu is considered the largest native oleifero resource in the world and one of the main forest products in Brazil [1]. The state of Maranhão is responsible for 94.5% of the whole national production of this native palm tree. The total production of babassu kernel in Brazil, was 83,917 tons in 2013 [2].The average composition of the babassu endocarp is 59%, summing up a residue of 707,300 tons in 2013. The majority of this rich mass lignocellulosic residue is improperly disposed, impacting negatively on the environment. The potential of babassu is numerous, however, in spite of its potential, only charcoal and oil have been produced in commercial scale [3]. The babassu nutshell showed high basic density and suitable lignin content for the sustainable production of bioenergy and

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charcoal, and is technically capable of replacing coke in Brazilian steel plants [4]. This can contribute decisively to the economic development of extractive communities, who survive from the babassu nut collection by marketing a product with higher added-value. [5].

The babassu carbon study has prioritized its use to provide power to department such as: steel industry, metal recycling and household use. Considering that all bibliography consulted showed work produced in conventional oven, there is a blank that must be filled in this research area, as well as a need in minimize outlay and in find out others products. Thus, as example, the production of babassu activated carbon using microwave as power supply. The use of the residues in preparation or production of a higher valued material as an alternative transforming waste in raw material. Recently, many studies have reported the production of activated carbon (AC) from residues such as: Rice husk, [6]; Green Coconut Shell,[7];Babassu coconut,[8];Mukah coal, [9]; Residues of babassu, [10]; Coffee waste, [11];Oil palm, [12], etc. Many studies have discussed great potential of lignocellulosic as a renewable feedstock for preparation of Activated Carbon. [13, 14].

Activated carbon (AC) is a carbonaceous and porous material prepared from the carbonization and activation of organic substances, especially from vegetable source. It is widely used in pollutant adsorption in liquid and gas phases, as catalyst support, to purify various compounds, to treat effluents, etc. The adsorption capacity of AC depends on the nature of its precursor, size of its particles (granules or powder) and on the production process chosen. The AC quality is evaluated in terms of its physical properties of adsorption and superficial area [15]. The success of activated carbon among these areas is mainly due to its extensive surface area and diverse chemical functionalities that can be modified during and after its production process.

The use of lignocellulosic material to produce activated carbon has been of interest to numerous researchers since it represents a “green” alternative for producing the carbonaceous adsorbent used all over the world. Also by using waste biomass as an activated carbon precursors, we not only take advantage of the availability and low cost of these materials but also raise an alternative for the treatment and disposal of hundreds of tons of biomass waste generated worldwide, which in most cases leads to a negative environmental impact, [16].

Microwave irradiation has recently exhibited superior performance for some organic chemical transformations, when compared to conventional heating methods. This is related to the high efficiency of microwaves in rapidly heating media[17]. The microwave heating has huge potential as effective method in order to produce activated carbon with high quality, high carbon yield and porosity[18]. In addition, the pores of activated carbon produced via microwave heating are uniform and clean. The BET surface area of activated carbon is still high. The eco-friendly activated carbon prepared via microwave heating can save significant energy and time activation [19].

In this study, the microwave irradiation system was used to produce babassu endocarp activated carbon (BAC) using physical activation with carbon dioxide as an activating agent. The properties of the activated material, such as surface area, pore size and yield were analyzed for the first time for conditions presented.

2. Material and methods

2.1. Starting raw material

In this work, endocarp babassu was selected as the starting raw material. The babassu fruit presents an ovoid or ellipsoidal shape with average length of about 10.00 cm, diameter of 6.80 cm and a weight of 273.00mg, as shown in figure1. The fruit has 4 constituents: externally, it has the epicarp a thin peal that involves the mesocarp this is a starchy secondary layer, and the endocarp this is a hard wooden, which accommodation the kernels.

Figure 1. Babassu with 2 and 5 kernels.
Longitudinal and transversal section.

The babassu used in this workwere collected in the rural area of Caxias city, in the state of Maranhão, Brazil (04º 51' 32" S, 43º 21' 22" W) and comes from the extractive activity of local communities. Only the endocarp received and dried in the atmospheric ambient of the sun was used to make the activated carbons. The endocarp was first crushed to the size fraction of 5.0-8.0mm; the particle size plays an important role in microwave heating because the penetration depth becomes significant as the particle size increases[20]. 50 g of babassu endocarp were used in each experimental run. Pyrolysis of the pre-treated endocarp was performed in a system consisting of modified multimode domestic microwave, and commercial coconut-based activated carbon (CAC) used as microwave absorber in the pyrolysis process was obtained from Laju group of companies, Malaysia.

2.2. Carbonization process

Carbonization was carried out in a microwave system consisting of modified multimode domestic microwave (800W, 2500 MHz), in a glass quartz reactor with a length of 20 cm and an inner diameter of 5 cm with bottom wire mesh, which serves as screen support as well as distributor plate. N_2 and CO_2 gases, K-type thermocouple (0 – 1200^{o}C range) were inserted into the bed region and data acquisition system was linked to personal computer for continuous monitoring and recording of process temperature[21-22]. For the pyrolysis of biochar, a single layer arrangement of precursor and coconut activated carbon (CAC) in the reactor was adopted because microwave heating requires the presence of microwave absorber to increase heat absorption. The size of CAC ranges from 1 mm – 1.8 mm. Thebabassu/CAC ratio was fixed at 1:0.5, irradiation time was 42min, with a heating rate of 50^{o}C/minand 30 minutes of residence time. The final temperatures were 600°C. A significant increase in the mechanics of biocharis achieved by carbonization at a final temperature above 500°C [23]. The choice of residence time and carbonization temperature was obtained from previous study of biochar babassu characterization especially for the preparation of activated carbons. The experimental results showed that it was feasible to prepare biochar with sufficient densities and relatively high porosities for preparation of activated carbons the best retention time was 30 min. Endocarp babassu biochar depends on the pyrolysis conditions and indicated that high retention time might affect the biochar characteristics. The carbonization temperature of 600^{o}C, i.e., the temperature at which the matter should be totally pyrolyzed, was possible by taking into account the thermo gravimetric curves of all raw materials used in this study. The microwave power of 800 W was selected for the pyrolysis. CAC was uniformly distributed on the wire mesh followed by babassu. N_2 was supplied at a flow rate of 400cm^3/min for about 5 min before commencement of experiments to ensure inert environment, and was reduced to 200cm^3/min during experiment so as to maintain the inert environment and to sweep out the vapor. After carbonization, the yield in biochar was calculated by dividing the dry mass of biochar by the dry mass of babassu endocarp multiplied by 100.

2.3. Activation process

The activation process was carried out in the same setup used for carbonization. All endocarp babassu biochar from the pyrolysis process were subsequently activated under the same conditions to prepare the final product. The activation temperature used were 700, 750 and 800^{o}C.The temperature was raised at a rate of 50^{o}C/min from room temperature to the desired activation temperature. During the

heating process, the nitrogen flow rate was 200 cm^3/min. Once the final activation temperature was reached, the gas supply to the reactor was changed to carbon dioxide (99.998%).The CO_2 flow rate used for the activation process was 200 cm^3/min and continued for 30 min. After activation, $N_2$was used again as an inert flushing gas whilst the microwave was allowed to cool down to room temperature.

2.4. Characterizations of raw babassu,biocharand activated carbon
Proximate analysis was carried out by a thermogravimetric analyzer – TGA before and after carbonization. Ultimate analysis was used to determine the carbon, hydrogen, nitrogen and sulfur contents of babassu and biochar using an Elemental Analyzer (Vario Micro Cube). Scanning Electron Microscope – SEM was used to verify the presence of porosity on the raw material and biochar surfaces.

2.4.1. Proximate and ultimate analyses. The thermal degradation studies of the raw material endocarp babassuwere conducted on Shimadzu DTA-50 analyzer, with heating rate of 10^{o}C/min from room temperature to 900^{o}C under N_2 atmosphere (20 ml/min).Proximate analysis of Biochar precursor and activated carbon was performed in the Thermogravimetric analysis - TGA (Pyris 6-TGA) to determine the amounts of moisture, volatile matter, fixed carbon and ash content in activated carbons TGA was used to follow the thermal degradation profile of the material after activation. About 20mg of sample were heated from room temperature at 30°C to 100°C in nitrogen until complete dehydration was accomplished, followed by decomposition at 600°C for 7 min to determine the quantity of volatile matters. The atmosphere was then changed to be oxidizing. The sample was cooled to 700°C, 750°C and 800°C and maintained at this temperature until its weight remained unchanged. The weight loss during this period was due to the reaction of the fixed carbon with oxygen and the remaining residue was ash.

The quantification of the carbon (C), hydrogen (H), nitrogen (N) and sulfur (S) relative to raw material and biochar was performed in a universal brand Elemental Analyzer (Vario Micro Cube). The oxygen content was determined by difference. The molar ratios H/C, N/C and O/C were also determined.

2.4.2. Scanning Electron Microscopy – SEM. Scanning electron microscopy (SEM) analysis with Jeol VPSEM IT300 was carried out for babassu,biochar and activated carbon to study the development of surface morphology and porosity.

2.4.3. BET – Surface Area. The physical properties of the biochars and activated carbons relating to specific surface area and total pore volume were obtained by measuring their nitrogen adsorption-desorption isotherms at -196°C in a surface area porosimeter system (Micromeritics 3Flex 3.01). Brunauer-Emmett-Teller (BET) equation was used to calculate the BET surface area.

2.4.4. Statistic analysis. The three-sigma rule was used for statistic determination of the results which states that even for non-normally distributed variables, at least 98% of cases should fall within properly-calculated three-sigma intervals[24].

3. Results and discussion

3.1. Raw Material – Babassu
The proximate and ultimate analyses of raw material are listed in table 1.The babassu endocarp presents a high content of volatile matter, a high content of fixed carbon and a low ash content, which are favorable properties for the preparation of activated carbon. The density of the babassu gives it the waterproof suggesting high lign in value so that the carbonization yield will be high. All properties of the babassu endocarp show that it is good precursors for activated carbon.

Table 1. Main characteristic of the raw babassu.

Characteristics	Raw material
Proximate analysis (wt %)	
Volatiles	63.39
Fixed Carbon	26.03
Ash	1.98
Moisture	8.60
Ultimate Analysis (wt %)	
Carbon	44.02
Hydrogen	5.87
Nitrogen	0.32
Sulfur	0.91
Oxygen[1]	48.88
C/N	137.56
H/C	0.13
O/C	1.11

[1]by difference

According to TGA results, to babassu endocarp raw material, the first degradation stage is related to water loss. From 220°C starts the second degradation stage which corresponds to the decomposition of cellulose and hemicellulose molecules. This thermal event presents maximum degradation speed close 400°C with mass loss of 53.02%. The third stage with mass loss 10.37% represents ash and fixed carbon contents. How, [25] says, the removal of extractives and lignin compounds affect the thermal stability of the materials, probably the increasing porosity.

The results show good agreement with [26] which found for volatile materials (83.40%) and a fixed carbon content (15.16%) in the babassu endocarp, being this more suitable for burning and carbonization, in relation to the other constituents of the fruit. The hemicellulose decomposes at 200–260°C, cellulose at 240–350°C, and lignin at 280–500 °C[27]. Therefore, for the first peak at 280°C in the rate of weight loss curve in figure 2, this weight loss is mainly due to the combined decomposition of hemicellulose and cellulose. The decomposition of both cellulose and lignin contributes to the subsequent peak at 350 °C.

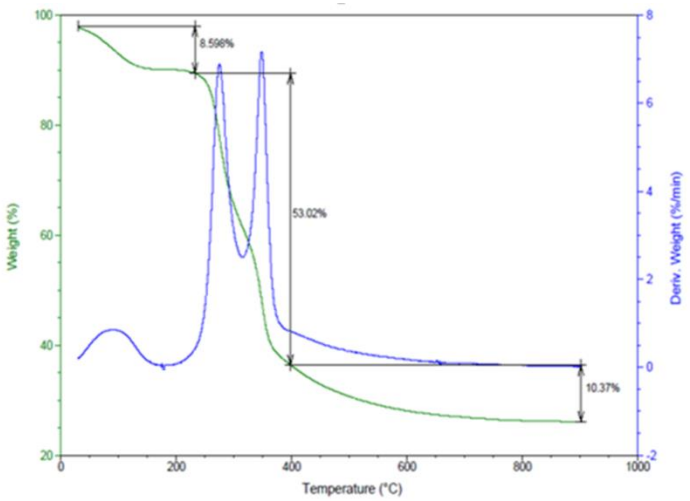


Figure 2. TGA curve for rawbabassu.

3.2. Temperature profiles

Pyrolysis temperature have significant effect on the characteristics of the activated carbons, [28]. During activation time, monitoring of process temperature was realized. The temperature ramp shown in figure 3, was obtained for all temperature from 700°C to 800°C. The feedback temperature control using microwave power is necessary for the precise temperature and the control in the reactor and thermocouples are the only available option[29]. It shows real time bed temperature profiles recorded during activation process of the AC. The cyclic ON/OFF of the magnetron in the multimode domestic microwave is responsible for the sinusoidal nature of the temperature profiles observed.

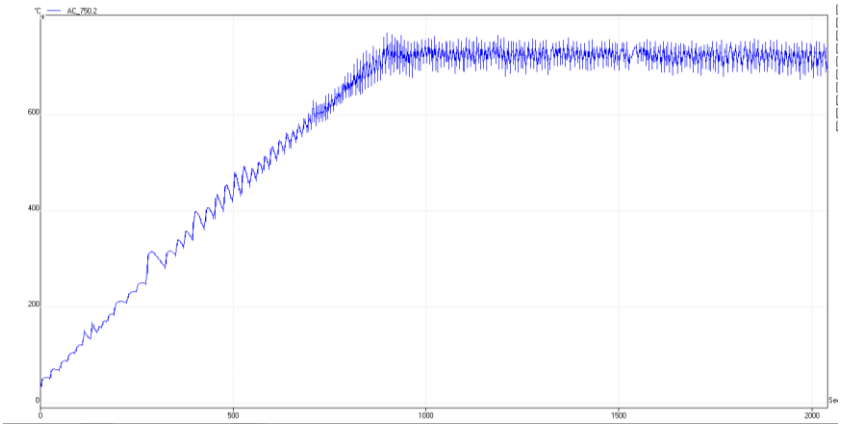


Figure 3. Temperature profile during MW activation of babassubiochar at 750 °C.

3.3. Thermogravimetric analysis of biochar and activated carbons

The Thermogravimetric analysis (TGA) profiles of the biochar and activated carbons are shown in figure 4. It is important indicator that all residues was burned and activated at 700°Cand have lower ash content. At 750 and 800°C the activated carbon have identical thermal properties.

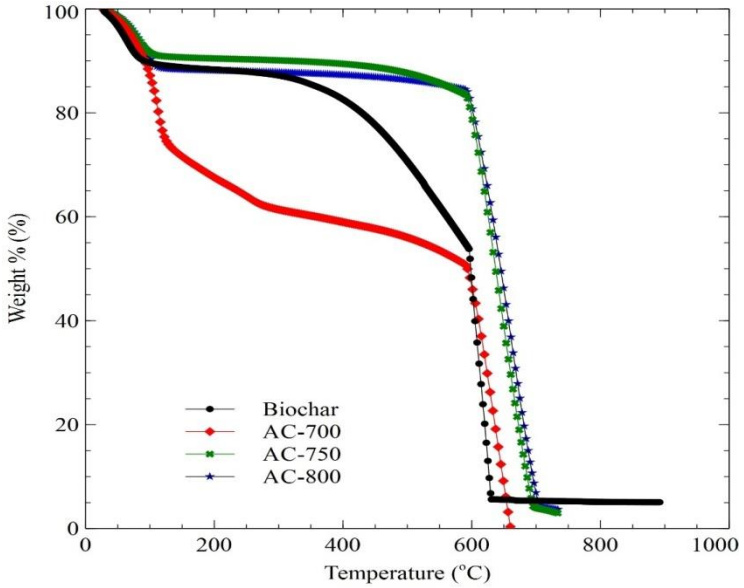


Figure 4. Thermogravimetric Analysis (TGA) of biochar and activated carbon.

The activated carbon produced under temperatures of 700, 750 and 800°C had its mass reduced in approximately 100°C (water lost), both showing begin of thermal decomposition around 340°C and stabilizing around 600°C, then, it can be assumed that the activated carbon produced, when compared to babassu biochar that originated it, have similar thermal stability . Another interesting aspect to be highlighted is regarding to the final mass. The babassu biochars resulted in 5.16% mass after stabilized, whereas the babassu in natural final mass was approximately 10.33%. AC_750°C biochar presented a residual mass around 2.8% and AC_800°C's was around 1.8%, whereas AC_700°C had no residual mass. The degradation process of lignocellulosic structures happened during all heating process according to [30–31]. It can be affirmed the material studied has an elevated lignin content in its solid matrix, once the amount of mass kept until temperatures of 400°C is considerably high. That condition is due to the fact that only the mesocarp of babassu coconut was used, i.e. it's most resistant part. Also should be emphasized the low level of ashes found in babassu activated carbon, which is an important property in choosing a good precursor for the activated carbon production. It is observed, as well, through the thermal analysis of activated carbon, a small amount of ashes after 700°C. These ashes suggest the presence of oxides or inorganic impurities formed after air heating. It is known that the presence of ashes can affect the chemical characteristics and the adsorptive behavior of activated carbon. This way, the low level of ashes found in the studied carbon is a desirable characteristic.

Table 2. Properties of Activated Carbons product at different temperature.

Properties	**Temperature (°C)**		
Proximate analysis (wt %)	**700**	**750**	**800**
Moisture	12.579	8.248	9.415
Volatiles	37.881	9.958	9.316
Fixed Carbon	62.119	78.926	79.509
Ash	-	2,867	1.760
Ultimate Analysis (wt %)			
Carbon	79.089	80.942	83.722
Hydrogen	2.279	2.120	1.623
Nitrogen	0.780	0.701	0.477
Sulfur	0.098	0.071	0.061
Oxygen[1]	17.753	16.166	14.118
C/N	101.376	115.496	175.655

[1]by difference

3.4. Yield of babassu endocarp biochar

The average yield for babassu endocarp biochars, as showed in figure 5, which passed through the pyrolysis process at 600°C with retention time 30 minutes, was 27, 66%. This value is similar to that obtained for the carbonization of babassunutshell25.8%[32] and shows the potential of fixed carbon production through the slow pyrolysis of babassu nut residues. The pyrolysis conditions are an important role in the development of the rudimentary pore structure in the biochar, through the release of volatiles from the carbon matrix. As this pore development in the biochar has a direct impact on the pore characteristics of the activated carbon the optimization of the pyrolysis parameters before activation is important, [33].

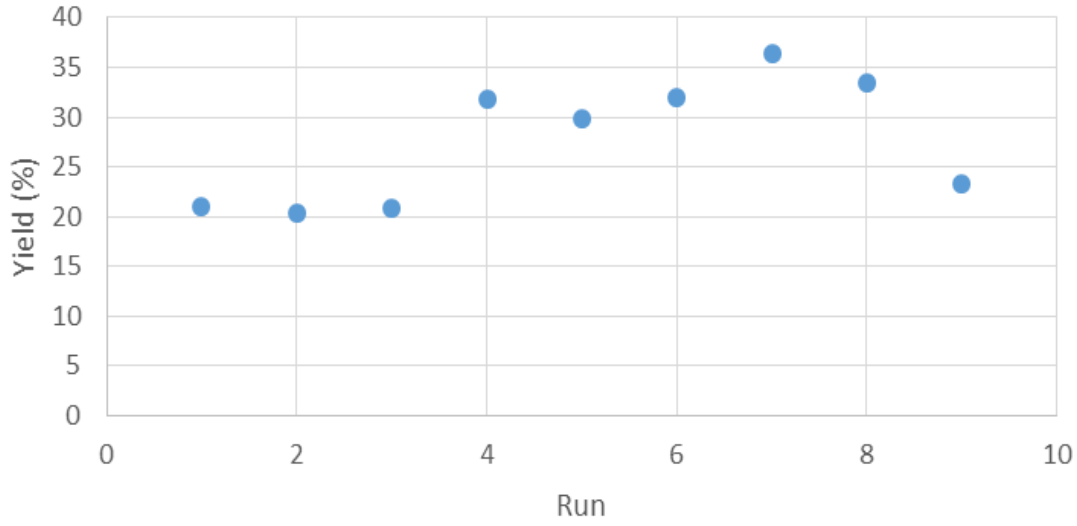


Figure 5. Yield of babassuendocarp biocharwith pyrolysis temperature at 600ºC and retention time 30 minutes.

This highlights the carbonization potential of babassu endocarp, considering the quality and yield of charcoal which, combined with the high density of this biomass, provide a biochar that meets the specifications needed for production activated carbon. The statistic of activation carbon relative babassu endocarp biocharare shown in table 3. This statistical component aimed to evaluate the heat treatment reproducibility, the possible variations presented by the Microwave and the system as a whole. It can also demonstrate the reproducibility of activated carbon itself, thus providing information on the babassu biomass characteristics used in the activated carbon production.

Table 3. Yield Statistic Activate Carbon

Expected Percentage	Occurrence Interval	
68.27%	14.88816	17.02684
95.45%	13.81882	18.09618
99.73%	12.74947	19.16553

3.5. BET - surface area

BET surface areas of the AC pyrolyzed at 600°C and activated at 700, 750 and 800ºC as shown in table 4, indicates an activated charcoal with high surface area and the pore size of all the activated carbons prepared in this study are mainly microporous.

Table 4. BET results.

Activation Temperature(ºC)	BET surface area(m^2/g)	Total pore volume(cm^3/g)	Micropore volume(cm^3/g)	Pore Size(nm)
700	543.5550	0.224879	0.190095	1.65
750	480.7335	0.188455	0.173761	1.57
800	542.1852	0.226877	0.192316	1.67

The microporosity found in the activated carbon from the Babassu endocarp done in microwave agrees with the results obtained from babassu AC produced in conventional oven. The difference in the values obtained are probably due to different conditions of HTT and activation used by researchers and also due to the use of three components of the shell together i.e., epicarp, mesocarp and endocarp,[34-35].This development of microporosities in activated carbons prepared from oil palm stones points to potential applications in gas-phase adsorption for air pollution control,[36] and the granular activated carbons from babassu had capacities to adsorb iodine, methylene blue, and residual chlorine, [37]

The figure 6 show the adsorption and desorption isotherms of activated carbons produced at 700, 750 and 800°C. All the N_2adsorption isotherms at 77K of the activated carbons show similar shapes. In isotherm curves, most nitrogen uptake occurs at the initial steep part until a relative pressure of 0.1. This adsorption type is called a type I isotherm, which reveals that the activated carbons produced were a microporous material, [38-39].

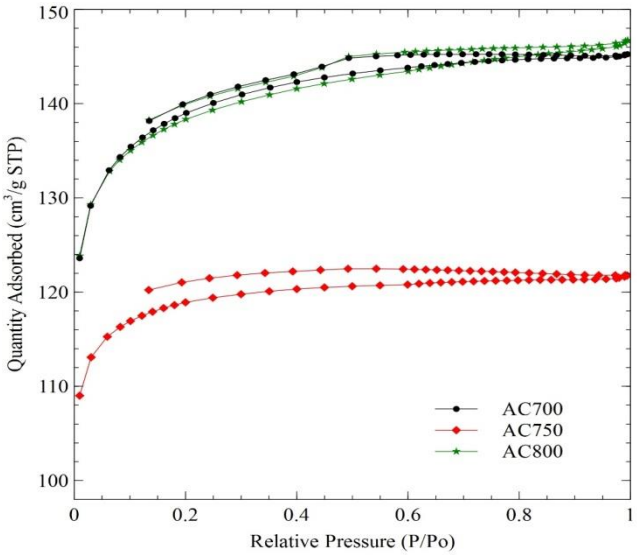
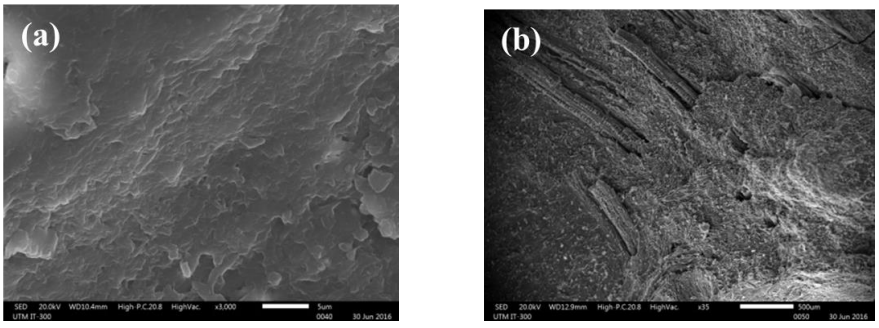


Figure 6. Adsorption–desorption isotherm of N_2 at 77 Kof the activated carbons at 700, 750 and 800°C.

3.6. SEM micrograph

The pore characteristics of the different activated carbonsstudied were analyzed using Scanning Electron Micrographs - SEM. Clear differences in porosityand particle shape were observed, and representative images areshown in figure 7. These images also show characteristics of babassu endocarp and the particlesthat formed during thebabassu endocarp pyrolysis and activation and their differentphysical characteristics.



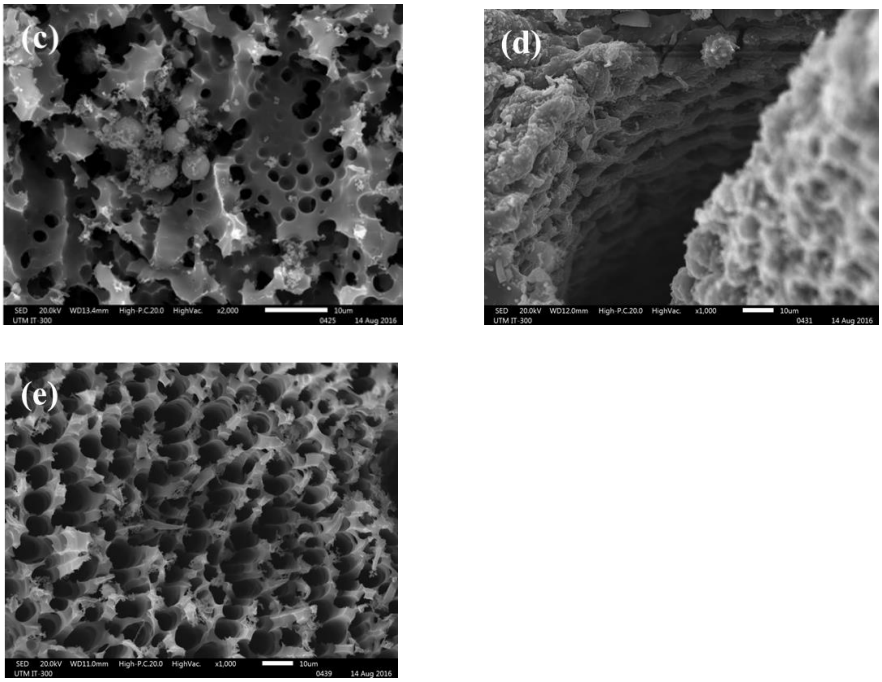


Figure 7. SEM images of(a) raw babassu endocarp, (b) biochar carbonized at 600^{o}C, (c) AC-700, (d) AC-750,and (e) AC-800.

Scanning Electron Micrographs - SEM realized on raw babassu endocarp, babassu endocarp biochar spyrolyzed at 600^{o}C with retention time 30 min and babassu endocarp activated Carbon indicatethat the initial structure of the raw material (figure 7a) shows a dense surface without pores. The pyrolysis creates a lotporosity (figure 7b). There are small "black spots" in lamellar structure of AC (figure 7c). These "black spots" are pores, perhaps micropores because pores are formed and destructed simultaneously during the activating period [40].The comparison of figure 7c, 7d and 7e indicates clearly that a regular porosity and a rather homogeneous surface was obtained by activation at 800^{o}C. These indicate that there were many orderly pores over the surface, forming a system of advanced pore structures.

4. Conclusion

This research shows that endocarp babassu can be used as precursor to produce activated carbon with a rather well-developed porosityby pyrolysis and physical activation by two-steps with CO_2activation via microwaves radiation. The yield statistic of activated carbon is within the desirable. SEM examination of raw material, pyrolyzed endocarp babassu and produced activated carbons shows that the porosity increases gradually after pyrolysis and activation. Nitrogen adsorption shows that the activated carbons obtaine dare essentially microporous with an average BET surface area.The temperatures of 700 and 800°C produce AC with similar BET, and the temperature of 750°C produces AC with intermediate BET

In summary, the activated carbon with a low production cost, this carbon material could be suitable for applications in gaseous pollutant adsorption, adsorbent of iodine, methylene blue and residual chlorine.

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