

RESEARCH ARTICLE

BIOCHARS FROM SLOW PYROLYSIS OF PEANUT SHELLS, CASHEW NUT SHELLS, AND MILLET STALKS AS VALUE-ADDED PRODUCTS FOR BRIQUETTE PRODUCTION

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Manuscript Info

Abstract

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Biomass is nowadays used for producing biochar briquettes through slow pyrolysis technologies namely for cooking purposes in developing countries. This article investigated some properties of biochar obtained from tropical biomasses like peanut shells, cashew nut shells, and millet stalks, pyrolyzed at 400 °C and 800 °C. Firstly the mass yield and energy yield of the biochar were determined. The main properties investigated were obtained by conducting proximate and ultimate analysis, determination of calorific value, FTIR (Fourier-transform infrared) analysis, and thermogravimetric analysis. The results showed that depending on the biomass and the pyrolysis temperature, the biochar yields varied from 20.26 % to 42.79 % while the energy yields varied from 24.24 % to 60.71 %. The highest yields are obtained with the lowest temperature and are all more important with peanut shells. All the biochars had fixed carbon contents greater than 60%, except biochar obtained at 400°C with millet stalks. The millet stalks biochar obtained at 400 °C was the one with the highest ash content (21.41%) and the lowest fixed carbon content (59.31%). It was observed that the more the pyrolysis temperature increased, the more the carbon content increased. The lower heating value of raw biomasses and biochars varied respectively between 18.36 and 22.51 MJ/kg and between 23.83 and 30.85 MJ/kg. FTIR analysis results showed that the O-H and C-O bonds disappeared in the case of biochars obtained at 800 °C. The reactivity of biochars towards O₂ showed that for all biochars, ignition temperatures (Ti) were up to 317 °C and the temperatures at maximum mass loss rate were between 438 °C and 501 °C.

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Introduction:-

In recent years, questions about the transition from fossil fuels to renewable energies have been mediated and debated by many states around the world. The conferences in Paris in France (COP 21, 2015) and in Marrakech in Morocco (COP 22, 2016) with the signing of the Paris agreements illustrate this awareness of politicians and decision-makers about the effects of climate change in connection with the use of fossil fuels.

Among renewable energies, biomass is one of the possible alternatives to the problems associated with the use of fossil fuels. In Senegal as in Africa, the use of biomass as an energy source is mainly dominated by two fuels: firewood and charcoal. However, the use of these two solid fuels is proving to be problematic, with the consequence

of deforestation and the emission of toxic pollutants (CO, PM2.5, NOx, etc.) harmful to human health when used in basic burning devices. Statistical data from the World Health Organization indicated that 7 904 deaths recorded in Senegal in 2016 were linked to indoor air pollution related to household cooking with solid biomass fuels (WHO, 2018). According to the 2017 FAO report, global charcoal production will continue to increase in the coming decades due to population growth, poverty, urbanization, and the relatively high prices of alternate energy sources for cooking (FAO, 2017). That means this situation will contribute to deforestation.

To reduce the environmental and health problems linked to the use of these two solid fuels, one of the possible alternatives is the sustainable production of quality biochar briquettes from non-upgraded biomass (mainly agricultural residues). One of the technologies relevant to this alternative is pyrolysis.

Among the array of biomass conversion technologies, pyrolysis is a relatively simple technique in which organic material is heated in the absence of oxygen [1]. During the pyrolysis process, three products are in permanent competition. These products are the char (solid fraction), the liquid (bio-oil), and the non-condensable gases. The pyrolysis process is conducted under very low oxygen concentrations, at pyrolysis temperatures ranging from 300 to 1100 °C, using variable residence times (seconds to hours) [2]. Depending on the operating conditions, the pyrolysis process can be subdivided into two classes: slow pyrolysis and fast pyrolysis. The terms "slow pyrolysis" are not precisely related to the time and speed of heating; they are sometimes arbitrary [3].

According to the studies of Balat et al., [4] and Bridgwater [5], generally slow pyrolysis occurs when temperatures range from 300 to 700 °C, the heating rate from 6 to 60 °C/min, and residence time from 300 to 1800 s. For fast pyrolysis, temperatures range from 600 to 1000 °C, the heating rate from 600 to 1200 °C/min, and residence time from 0.5 to 10 s. The main product obtained during slow pyrolysis is the solid fraction while in the fast pyrolysis process, the liquid fraction (bio-oil) is the dominant product. Many studies in the literature are focused on slow pyrolysis [6], [7] or fast pyrolysis [8], [9].

This study aims to determine the physicochemical characteristics of biochars for their use as a source for producing biochar briquettes by using different pyrolysis conditions (slow pyrolysis) and some agricultural wastes used as raw biomass. To achieve this objective, some physicochemical properties of biochars obtained at different pyrolysis temperatures (400 and 800 °C) were evaluated.

Materials and Methods:-

Raw materials

Three biomasses produced in Senegal and considered agro-residues were used in this study. The biomasses were peanut shells (PNS), cashew nut shells (CNS), and millet stalks (MS). Peanut shells and cashew nut shells were collected respectively from the SONACOS company (Ziguinchor/Senegal) and a cashew nut processing unit (Ziguinchor/Senegal). Millet stalks came from agricultural farms.

Before pyrolysis, millet stalks were cut into small pieces less than 5 cm in length and then peanut shells and millet stalks were dried below 2 % moisture content. The operation of drying biomass before pyrolysis was not applied in the case of cashew nut shells due to the presence of cashew nut shell liquid.

These three biomasses were selected depending on their energy potentialities and their large availability in the Ziguinchor area. Table 1 shows some characteristics of these biomasses and their availability per year during the period 2014 to 2019. Among the three biomasses, peanut shells had the highest fixed carbon (FC) content. But the lower heating value (LHV) was more important for cashew nut shells. Large amounts of millet stalks and peanut shells were generated based on the FAOSTAT (Food and Agriculture Organization Corporate Statistical Database) data.

Samples	$\operatorname{Ash}(\%)^{\mathrm{a}}$	FC $(\%)^{a}$	LHV (kJ/kg) ^a	Quantity (tones/an)
PNS	9.86	20.78	18.48	524 230
CNS	2.60	15.80	21.92	6113
MS	9.49	14.88	18.12	1 852 247
References	[10], [11]	[10], [11]	[10], [11]	Estimation based on FAOSTA Data
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Table 1:- Literature	data	of the	three	biomasses.
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^a: expressed on a dry basis

Pyrolysis process

Biomass pyrolysis experiments were conducted in an externally heated fixed bed reactor (see Figure 1). The electrical reactor is the same as that used by Dufourny et al., [12]. The main objective of these experiments was to produce biochar with fixed carbon content above 60 %.

Two pyrolysis temperatures were selected: 400 °C and 800 °C. The biomass samples were pyrolyzed with a heating rate of 5 °C/min and the time at final temperatures was 2 hours. Due to the apparent density difference between the three biomasses, about 6 to 6.5 kg of peanut shells, 3 to 8 kg of cashew nut shells, and 2 to 3 kg of millet stalks were used during the pyrolysis tests. The reactor was heated by a tubular resistance furnace with a power of 20 kW consisting of three independent heating zones; which allows homogeneous heating of the biomass bed (peanut shells or cashew nut shells or millet stalks).

A preheated nitrogen flow of 10 L/min was injected into the reactor through a steel coil to flood the atmosphere and expel volatile vapors. The pyrolysis gases are discharged from the top of the reactor through the gas exhaust pipe and are then burned in the post-combustion chamber to prevent any external air pollution. During the cooling phase, the air inlet was connected to a blower to accelerate the cooling of the reactor by convection.



Figure 1:- Image of the Fixed bed reactor.

Determination of mass yield and energy yield of biochars

The mass yield (Y_{char}) , and energy yield (η_E) of biochar were defined as the following equations.

$$Y_{char} (\%) = \frac{M_{char}}{m_{bio}} \times 100$$
(1)

$$\eta_{E} (\%) = Y_{char} \frac{HHV_{char}}{HHV_{bio}}$$
(2)

 m_{char} is the mass of biochar (dry basis), m_{bio} is the mass of raw biomass (dry basis). HHV_{char} is the higher heating value of the biochar and HHV_{bio} is the higher heating value of the raw biomass.

Characterization of raw biomass and biochars

The analyses conducted in this study concern the proximate analysis, the ultimate analysis, the calorific value determination (HHV and LHV), the FTIR analysis, and the thermogravimetric analyses.

The proximate analysis conducted in a muffle furnace was based on the NF EN 1860-2. XP CEN/TS 15148 and XP CEN/TS 14775 norms to determine volatile matter content (VM) and ash content (Ash). For the determination of volatile matter content, the biomass or the biochar was heated without air at 900 °C for 7 min. Ash content was

determined after combusting biomass at 550 $^{\circ}$ C and for the biochar at 710 $^{\circ}$ C for 2 hours. Fixed carbon content (FC) was derived from the difference.

$$FC (\%) = 100 - VM (\%) - Ash (\%)$$
(3)

Moisture content (M) was determined in an oven at 105 °C during about 3h of drying following the AFNOR NF EN 1860-2 norm.

Elemental analysis was performed by using an elemental analyzer (VarioMACROcube) following ASTM D5373 and XP CEN/TS 15104 norms. Carbon (C), hydrogen (H), and nitrogen (N) contents were determined and the oxygen (O) content of the sample is obtained by difference.

For the determination of the carbon, hydrogen, and nitrogen contents, the solid samples are finely ground and homogenized (1 mm for the raw biomass and 200 μ m for the biochar). The sample (mass weight < 100 mg), placed in tin foil, is introduced into an oven at about 960 °C in a flow of oxygen (130 mL/min for biochar and 100 mL/min for biomass). The combustion of the sample produces the following molecules: CO₂, H₂O, NOx, SO₂, and SO₃ as well as volatile halogen compounds. Entrained by a flow of helium (600 mL/min), all of the molecules pass through the reduction tube. The NOx is then reduced to N₂ via the tungsten. Volatile halogens and excess oxygen are trapped by silver wool and copper, respectively (creation of AgCl and CuO₂). The sulfur products are trapped in the tungsten forming metallic W-S bonds. At the exit of the reduction tube, the molecules present in the helium flow (carrier gas) are N₂, CO₂, and H₂O. These molecules are directed to the thermal conductivity detector. The CO₂ and the H₂O are trapped on a molecular sieve while the nitrogen passes through the detector and causes a variation in conductivity which materializes thanks to the software by the recording of a peak that can be integrated to deduce a quantity of nitrogen measured.

Once the nitrogen quantification is complete, the CO_2 molecules are desorbed (temperature rise of the molecular sieve) and analyzed in turn by the detector. When the CO_2 quantification is complete, the molecular sieve holding H_2O is in turn heated to release the H_2O molecules which are in turn quantified.

The element concentration was computed from the detector signal, and the sample weight was based on stored calibration curves.

The analysis for determination of the higher heating value (HHV) on a dry basis was performed in a calorimeter (model Parr 6200) following the XP CEN/TS 14918 norm. The lower heating value (LHV) on a dry basis was calculated using the following equation [13].

 $LHV (KJ / kg) = HHV - 212.2 \times H$ (4)

where H is the hydrogen content in the percentage of the sample.

The FTIR analysis spectra were obtained for both raw biomass and biochar by using a frontier FTIR spectrometer (model PerkinElmer). The different spectra were baseline corrected and averaged using the Spectrum v10 software (PerkinElmer Spectrum). Data from spectra were extracted in CSV files and then they were retraced in OriginPro Ver 9 software.

For evaluating the different characteristics of the combustion of biochars, a thermogravimetric analysis (TGA) was performed in the O_2 atmosphere. The thermogravimetric analyzer used in the study is not purchased. It was developed by the CIRAD experts (see figure 2). It is composed of a rubotherm type magnetic microbalance, a 25 mm diameter quartz reactor, an electric furnace, and type K thermocouples. The temperature of the microbalance is set constant at 24 °C due to a cooling system with water circulation via a cryostat. Heating rates and temperatures in this device can reach 20 °C/min and 1000 °C respectively. The device measures both the loss of mass and the variation in temperature over time. Two gas cylinders (N₂ and CO₂) were connected to the reactor by flowmeters. Two Brooks 5850 S flowmeter types, calibrated for N₂ and CO₂ in an interval of 0 to 400 Nm³/min were used. The pressure inside the reactor is controlled by a Brooks 5866 brand pressure controller. It takes pressure ranging from 0 to 150 bar. To limit systematic measurement errors, the magnetic microbalance needs to be calibrated at each start of the test. The O₂ flow rate was set at 100 mL/min. Samples were heated from 20 °C to 1000 °C with a heating rate of 10 °C/min. When the temperature reaches 1000 °C, it is maintained in this stage for 1 hour. Sample masses used were 63.3 mg for peanut shells and cashew nut shells and 32.0 mg for millet stalks. Mass loss was recorded every five seconds and the mass loss rate was calculated. The FFT filter's function was applied to the mass loss rate curves

for smoothing.



Figure 2:- Picture of the thermogravimetric analyzer developed by the CIRAD experts.

Results and Discussion:-

Mass yield and energy yield

Table 2 presents the results of the calculations of the mass and energy yields of biochar, on a wet basis, obtained during pyrolysis of samples of peanut shells, cashew nut shells, and millet stalks in a fixed bed reactor.

Depending on the biomass and the pyrolysis temperature, the mass yields of biochars varied from 20.26 % to 42.79 % while the energy yields varied from 24.24 % to 60.71 %. The highest yields are obtained with the lowest temperature and are all more important with peanut shells. These results are comparable to those of Balat et al., [4]; Bridgwater [5], and Noumi [3] who found mass yields of biochar in the order of 20 to 49 % during the slow pyrolysis of different biomasses. The pyrolysis technology used seems to be efficient, especially for samples of peanut shells since Goyal et al., [14] and Park et al., [15] stated that for the most efficient pyrolysis processes, the mass yields of biochar which can vary depending on the composition of the biomass reach on average 30 % on a wet basis.

The energy yield remains higher than the mass yield whatever the biomass. This confirms that pyrolysis is a process that consists in concentring the energy content of given biomass. The energy yields of biochar at 400 °C of the three raw biomasses are between 45 % and 61 %, close to those found by Park et al., [15] in the case of pyrolysis of rice straw at 400 °C (around 50.5 % for a heating rate of 10 °C/min, with nitrogen flow of 1.5 L/min and maintaining time during 1h).

Characteristics	Temperature	PNS	CNS	MS
Biochar mass yield, Y_{char} (%)	400°C	42.79	33.93	37.62
	800°C	35.01	20.26	28.98
Biochar energyyield, η_E (%)	400°C	60.71	45.19	47.08
	800°C	47.42	24.24	43.42

Table 2:- Mass and energy yields result	ılts	resi	vields	energy	and	Mass	2:-	ble	Та
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Results of proximate and ultimate analyses

The results of proximate and ultimate analysis of the raw biomasses and biochars are given in table 3. For raw biomasses, peanut shells have the highest fixed carbon content with the highest ash content measured at 5.95 %. For the three raw biomasses, the results of the proximate analysis are to be found close to those of Ba et al., [16] and Tagutchou et Naquin [17]. Regarding the results of the ultimate analysis, the elemental compositions of peanut shells and millet stalks seem relatively close. Nevertheless, the elemental composition of cashew nut shells stands out a little with a relatively higher carbon content of 57.9 % against around 49 % for peanut shells and millet stalks. **Table 3:-** Results of proximate analysis.

Samples	Proximate analysis on a dry basis (%)				Ultimate analysis on a dry basis (%)			
	M ^b	VM	Ash	FC	С	Н	Ν	O ^a
Biomass								
PNS	9.35	70.75	5.95	23.30	48.85	5.57	1.40	38.23
CNS	8.91	82.54	1.99	15.47	57.76	6.71	0.48	33.60
MS	10.47	81.86	2.49	15.65	49.14	5.64	0.35	42.38
Biochars								
PNS_400	0.34	15.54	14.25	70.21	71.10	3.28	2.11	9.25
PNS_800	1.28	1.63	20.01	78.36	75.22	0.79	1.67	2.32
CNS_400	0.30	27.51	6.08	66.41	76.26	4.62	1.35	17.87
CNS_800	1.12	2.05	13.50	84.45	85.15	0.92	1.12	-
MS_400	1.27	19.28	21.41	59.31	62.15	3.09	0.74	12.62
MS_800	1.80	6.94	10.81	82.25	83.29	0.92	1.32	3.67

M: Moisture; VM: Volatile mater; FC: Fixed carbon; C: Carbone; H: Hydrogen; N: Nitrogen and O: oxygen; ^b: expressed in wet basis; ^a: obtained by difference.

By analysing the results of proximate analysis of the obtained biochars, we found that practically all the biochars (except biochar MS_400) have fixed carbon contents greater than 60 %. The biochar of millet stalks obtained at 400 °C was the one with the highest ash content (21.41 %) and the lowest fixed carbon content (59.31 %). For biochars, the results of the ultimate analysis indicated an increase in carbon content with the increase in temperature.

Higher heating value and lower heating value

Table 4 summarizes the results of the determination of the calorific values of all studied samples. This table shows that the lower heating values of raw biomasses and biochars varied respectively between 18.36 and 22.51 MJ/kg and between 23,83 and 30.85 MJ/kg. The lower heating values of raw biomasses are close to those found by Diedhiou [11] and Tagutchou and Naquin [17]. For biochars, the lower heating values are in the range of those found by Ábrego et al., [18] and Liu et al., [19].

Table 4:- Results of calorific values determination	on.
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Samples	Calorific value on a dry basis (MJ/kg)			
	HHV	LHV		
Biomass				
PNS	19.55	18.37		
CNS	23.93	22.51		
MS	19.56	18.36		
Biochars	· · · · · · · · · · · · · · · · · · ·			
PNS_400	27.74	27.04		
PNS_800	26.48	26.31		
CNS_400	31.87	30.85		
CNS_800	28.63	28.43		
MS_400	24.48	23.83		
MS 800	29.31	29.12		

Table 4 also shows a slight decrease in the calorific value of biochar of peanut shells and cashew nut shells by increasing pyrolysis temperature from 400 °C to 800 °C. By contrast, the calorific value of biochar of millet stalks increases with the increase of pyrolysis temperature. This behaviour is typical of biomasses with high ash content [20]. For biochars of cashew nut shells, the decrease in the calorific value can be also attributed to the release of CNSL (Cashew Nut Shell Liquid) which has a high calorific value.

Chemical group information

FTIR spectra of the three biomasses and their biochars were analysed based on the IR table (see http://lmspn.uqam.ca/fichiers/tablesir.pdf) and literature data. FTIR spectra are shown in figure 3. The wide peaks observed at 3315 cm⁻¹, 3294 cm⁻¹, and 3344 cm⁻¹, respectively for peanut shells, cashew nut shells, and millet stalks were attributed to O-H bond stretching vibrations. The C-H bond stretching vibrations indicating the presence of methyl and methylene groups in the structures of the three biomasses were observed at bounds between 2850 and 3000 cm⁻¹. Peaks observed between 1600 and 1650 cm⁻¹ were attributed to C=C bond stretching vibrations. Peaks observed at around 1000 cm⁻¹ are characteristics of those of C-O bond stretching vibrations indicating the presence of primary alcohol in the structures of the three biomasses. Similar structures were obtained during conducted studies on these three biomasses [21]–[23].

Figure 3 shows that pyrolysis has modified the structures of the three biomasses regarding the spectra of the biochars. The O-H bond begins to disappear in the case of obtained biochars and this, is as important as the final temperature of pyrolysis is high. The C-O bond characterizing the peaks observed at around 1000 cm⁻¹ on the FTIR spectra of biomasses has completely disappeared in the case of biochars obtained at 800 °C, but the intensity of the peaks is low in the case of biochars obtained at 400 °C. Thus, the increase of the final temperature of pyrolysis promotes the release of oxygen groups mainly with water form, carbon monoxide but also alcohols, acids, and aldehydes. These trends of the disappearance of oxygen groups were also remarked by [24], [25].



400 °C and 800 °C, respectively.

Biochar's reactivity to O₂

Biochar's oxidation was examined using thermogravimetric analysis. The results are shown in figure 4. The results showed that decomposition of biochars of millet stalks is done on a less wide band (between 200 and 600 [°]C) compare to those of biochars of peanut shells and cashew nut shells (between 200 °C and around 800 °C). Experiments done indicated that biomass pyrolyzed at high temperature (800 °C) showed peaks at maximum mass loss rate at relatively high temperatures of 503.87 °C, 467.78 °C, and 455.09 °C, respectively for biochars from peanut shells, cashew nut shells and millet stalks, probably due to the low content of volatile matter on the biochars. Boukaous et al., [26] found temperatures of 535 °C and 426 °C at maximum mass loss rates, respectively for Biochar of date kernels (at 850 °C) and biochar of Aleppo pin husks (at 850 °C).

On figure 4, we note that the more is the final temperature of pyrolysis, the more is the temperature at the maximum mass loss rate. This is probably due to the content of volatile matter in the biochar. For a given type of biochar, the more the volatile matter content, the less the temperature at the maximum mass loss rate.



Figure 4:- Curves of mass loss (m/m₀) and the mass loss rate (dm/dt) of all biochars.

Table 5 summarizes some combustion characteristics obtained from curves of the mass loss and the mass loss rate of all biochars. These characteristics are the ignition temperature (T_i) , the temperature at maximum mass loss rate (T_{max}) , the maximum mass loss rate $(dm/dt)_{max}$ and the average mass loss rate $(dm/dt)_{mean}$.

The ignition temperature was determined as indicated by [27]. For all biochars, ignition temperatures (T_i) were up to 317 °C. On average, the decomposition of biochars of cashew nut shells is quicker than those of the other biochars. Biochars of millet stalks present the highest values of maximum mass loss rate (4.66 for MS_400 and 4.19 for MS_800).

Table 5 shows that the ignition temperature increases with the increase of the final temperature of pyrolysis, except for the case of millet stalks. In addition, biochar produced at a high final temperature of pyrolyzing (800 °C) presents a lower average mass loss rate than biochar produced at a low final temperature of pyrolysis (400 °C) except in the case of millet stalks where a contrary effect is observed. The higher average mass loss rate of the biochar of millet stalks produced at 800 °C could be explained by its lower ash content.Furthemore, it was observed

that the biochars produced at the final temperature of 400 °C showed the highest maximum mass loss rates. Similar variations in the ignition temperature and the maximum mass loss rate were observed by Protásio et al, Xiong et al., [28], [29].

Biochars	T _i	T _{max}	(dm/dt) _{max}	(dm/dt) _{mean}
	(°C)	(°C)	(%/min)	(%/min)
PNS_400	327.90	464.27	2.62	0.54
PNS_800	363.75	500.87	2.25	0.51
CNS_400	317.38	437.95	2.71	0.61
CNS_800	355.98	467.78	2.26	0.58
MS_400	347.21	437.95	4.66	0.51
MS_800	345.45	455.09	4.19	0.55

Table 5:- Characteristics of biochars combustion.

Figure 5 shows how the ignition temperature and the temperature at maximum mass loss rate were obtained (example of oxidation of PNS_400). The temperature at maximum mass loss rate corresponds to the temperature at which it is observed the greatest mass loss rate.



Figure 5:- Determination of the ignition temperature and the temperature at the maximum mass loss rate.

Conclusions:-

In this study, the main properties of biochars derived from pyrolysis of three biomasses at 400 °C and 800 °C were investigated for their use as a source for producing biochar briquettes. The three biomasses studied were peanut shells, cashew nut shells, and millet stalks. A fixed bed reactor was used to conduct pyrolysis experiments to produce briquettes. The biochar analysis provided a significant decrease in the mass yield of biochar with the increase of pyrolysis temperature from 400 °C and 800 °C. The fixed carbon and ash contents increased by increasing the temperature of pyrolysis (except in the case of the pyrolysis of millet stalks).

Pyrolysis was also found to increase the lower heating value of the biomass with the increase of the pyrolysis temperature. Due to the high values of the lower heating value, the obtained biochars will be suitable as a source for producing biochar briquettes.

According to the analysis of the FTIR spectra, the increasing pyrolysis temperature caused the disappearance of O-H et C-O functional groups in the structures of biochars, due to the release of oxygen groups mainly with water form, carbon monoxide but also alcohols, acids, and aldehydes.

During oxidation, ignition temperatures up to 317 °C were obtained and the temperatures at the maximum degradation were between 438 °C and 501 °C. The reactivity of biochars towards O_2 showed that biochars with cashew nut shells were the most reactive.

Considering the high mass and energy yields of biochar from peanut shells, and in terms of economy (biomass collection, transport, pyrolysis process), it would be preferable to use peanut shells for producing biochar briquettes.

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