International Journal of Sustainable and Green Energy
2020; 9(2): 23-28
http://www.sciencepublishinggroup.com/j/ijsge
doi: 10.11648/j.ijrse.20200902.11
ISSN: 2575-2189 (Print); ISSN: 2575-1549 (Online)

Science Publishing Group

Research/Technical Note

Charcoal Yield and Fixed Carbone Content of Jatropha Seed Cake Pyrolysis: Effect of Preheating Temperature, Extraction Pressure and Seed Form

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To cite this article:

Nsah-ko Tchoumboué, Lontsi Meli Raoul Gilles, Tangka Julius Kewir. Charcoal Yield and Fixed Carbone Content of Jatropha Seed Cake Pyrolysis: Effect of Preheating Temperature, Extraction Pressure and Seed Form. *International Journal of Sustainable and Green Energy*. Vol. 9, No. 2, 2020, pp. 23-28. doi: 10.11648/j.ijrse.20200902.11

Received: March 7, 2020; **Accepted**: March 27, 2020; **Published**: April 23, 2020

Abstract: The pyrolysis characteristics of cake from whole, kernels and crushed jatropha seed (Charcoal yield, fixed Carbone content, mineral matter) were assessed. The pyrolysis process consisted in introducing into an oven, at 300°C for 15 minutes, a 25 ml ceramic crucible containing 5 g of cake (2% water content) coated in pre-weighed aluminum foil. The main results were as follow. The highest charcoal yield (75,76±1,53%) was obtained with cake from crushed seeds preheated at 100°C associated to the extraction pressure of 15000 pounds. The highest fixed Carbone (18,64%) was registered with biochar from kernels cake obtained at the preheating temperature/extraction pressure couple of 25°C-15000 Pounds. The highest mineral matter content (Mg, Ca, P and K) are recorded in charcoal from the kernel cake pyrolysis. Seed form significantly affected the cake yield and biochar yield. The pH of jatropha biochar (7,5-10) are alkaline which can be useful for acid soil fertilization in Cameroon.

Keywords: Extraction Conditions, Jatropha Cake, Charcoal Yield, Fixed Carbone Content, Mineral Matter

1. Introduction

The production of charcoal is one of the promising and recent technology used in rural areas of developing countries nowadays. For this purpose, wood biomass is the principal raw materials directly used [1, 2]. This type of practice is of great consequence which is sometimes disastrous to ecological resources (deforestation and climate change). In order to resorb these problems, another biomass with a large diversity must be used as an alternative to conventional wood. Seed oils [3] in general and Jatropha curcas seem to be one of the promising renewable and independent sources in rural areas [4].

Jatropha is a multipurpose small tree of the family of Euphorbiaceae found in the tropics and subtropics [5, 6]. Although the principal use of Jatropha curcas is for biofuel production, the residual cake should not be neglected. The cake can be valorized by thermochemically and biologically procedures [7]. It is then important to produce knowledges for efficient protocol of Jatropha energetic valorization.

Scientific literature has been published on the various factors to improve pyrolysis process (Gas, liquid and charcoal production). For the moment, industrial liquid and gas production are not of great importance for rural areas of developing countries. We then focus only on charcoal issue. Notably the effect of particle sizes [8], chemical (cellulose, hemicellulose, lignin and lipids) composition [9], heating temperature and heating time [10] are the mains factors affecting biochar yields. Studies have analysed the pyrolysis of Jatropha cake under different particle size, heating temperature and residence time [11-13]. In fact, [14] found a charcoal yield from jatropha cake of 81,5% during hydro pyrolysis at 40 bar hydrogen pressure. No others information is given concerning the fixed carbon and the type of cake. Whatever the case, various extraction conditions are still be testing to optimize oil production. Thus, the characteristics of the produced cake can also affect the pyrolysis process. Many research deal in the contrary with the completely de-oiled cake without taking into account the extraction procedure. That's why the present topic is aimed to study the effect of cake, from the seed form preheated at different temperatures under different applied pressures, on the pyrolysis characteristics (charcoal yield, fixed carbon content and minerals composition of biochar)

2. Materials and Methods

2.1. Obtention of Jatropha Whole, Kernels, Crushed Seeds and Cake

Jatropha seeds used for the study were obtained from local neighboring farms. The whole seed was used as such. While the kernel was obtained using a manual rotary disk machine to dehull whole seed and crushed seeds (3.7 mm size particle) was obtained using a 2 kWh electric motor grinding machine. The different type of seeds each (whole, kernels and Crushed) were preheated at four levels of temperature (25°; 50°; 75° and 100°C) respectively combined to three following levels of extraction pressure (8400; 15000 and 19500 pounds). Each type of cake, obtained as described above, were weighed, packaged in the plastic bags and stored in an oven at 30°C for two weeks. Their bromatological characteristics (dry matter, volatile matter, residual lipids and cellulose) were determined according to [15]. After this, they were then submitted to the pyrolysis process.

2.2. Cake Pyrolysis

The pyrolysis process of each of the above type of cake was carried out using the method proposed by [2]. This method has the particularity to use small quantities of samples. An adiabatic "Heraeus" electric furnace was used at this purpose. For the pyrolysis experiments, 5 g of each type of cake (2,0% water content) obtained as mentioned above was coated in pre-weighed aluminum foil and introduced into a 15 ml ceramic crucible. Aluminum foil has the ability to protect the samples from the oxidizing atmosphere. The nascent volatiles during pyrolysis could escape easily so that the pressure in the samples remained practically equal to the ambient value. The crucible was then introduced into the furnace. One isothermal pyrolysis level was fixed at 300°C with a residence time of 15 minutes [16]. When the required temperature and the residence time was observed, the furnace was stopped, the sample removed, introduced into the desiccator and allowed to cool (25°C). The weight of the aluminum foil was subtracted from the weight of the sample still coated in aluminum foil in order to determine the exact weight of the biochar. After this, the biochar was removed from the package, introduced into the 12 grams plastic bags, labeled and stored in an oven to determine the fixed carbon and mineral content.

For the volatile matter content, 1g of charcoal was introduced to crucible and placed in a furnace at 550°C for one hour. The ash content was determined by heating the carbonized charcoal in an open crucible at 550°C for 4 hours. The AOAC method was used to determine the mineral elements (phosphorus, potassium, magnesium and calcium).

The biochar yield and the fixed carbon content was determined using the following equations:

$$Yc = \frac{Mc}{Mw} \quad (1)$$

$$Fc = 100 - Ash\ content - Volatile\ matter\ content \quad (2)$$

Yc: Biochar yield (%); Mc: Mass of char (grams); Mw: mass of cake (grams) Fc: fixed carbon content (%)

3. Results and Discussion

3.1. Cake Yield as a Function of Seed Type, Preheating Temperature and Applied Extraction Pressure

Independently of the seed type, the applied extraction pressure and the preheating temperature, the significantly highest cake yield (Table 1) was obtained with crushed seeds preheated at 50°C associated to the applied extraction pressure of 8400 pounds and the lowest yield with the kernels preheated at 100°C under the highest extraction pressure (19500 pounds). Whatever the seed form and the preheating temperature level, the cake yield decreases logically with increasing applied extraction pressure. The same observation was obtained with the preheating temperature whatever the seed form and the applied extraction pressure. Investigation on factors affecting these characteristic are not yet documented in the literature, indeed the smallest particle size in crushed seeds compared to whole and kernels, can be the main reason of the highest cake yield observed.

Table 1. *Cake yield as a function seed type, preheating temperature and applied extraction pressure.*

Seedforms	Extraction pressure	Preheating temperature			
	(Pounds)	25°C	50°C	75°C	100°C
Whole seeds	8400	86,67 ± 0,51bA*	82,93 ±3,59bA*	84,03 ± 2,68bB*	81,90 ± 0,58bC*
	15000	76,33 ± 0,51bA**	76,10 ± 1,25bA**	76,53 ± 0,60bB**	75,23 ± 1,55bC**
	19500	75,03 ± 0,85bA***	73,63 ± 1,70bA***	72,40 ± 0,70bB***	71,80 ± 2,36bC***

Seedforms	Extraction pressure (Pounds)	Preheating temperature 25°C	50°C	75°C	100°C
	8400	70,53 ±0,51cA*	69,63 ±3,59cA*	66,90 ±2,68cB*	61,33 ± 0,58cC*
kernels	15000	60,63 ±0,51cA**	62,10±1,25cA**	56,10±0,60cB**	54,50 ± 1,55cC**
	19500	56,10 ±0,85cA***	59,03 ±1,70cA***	54,63 ± 0,70cB***	51,50 ± 2,68cC***
	8400	88,36 ± 0,32aA*	89,26 ±0,80aA*	88,20±1,21aB*	84,80 ±0,26aC*
Crushed seeds	15000	81,40 ± 0,69aA**	83,30 ± 1,30aA**	83,00 ± 3,55aB**	78,26 ±0,66aC**
	19500	77,53 ±0,25aA***	77,20 ±1,76aA***	74,80 ± 0,80aB***	76,06 ±1,79aC***

a, b, c: means with the same letter on the same column are not significantly different (p>0,05)
A, B, C: means with the same letter on the same line are not significantly different (p>0,05)
*: means with the same number of star in the same column are not significantly different (p>0,05)

3.2. Effect of Seed Form, Preheating Temperature and Extraction Pressure on Cake Characterization

3.2.1. Volatile Matter

Regardless of the preheating temperature, extraction pressure, and seed shape, the significantly highest volatile matter (figure 1) was obtained with the cake from the whole seed extraction preheated at 100°C associated at the applied pressure of 19500 pounds while the lowest was registered with the non-preheated kernels at 25°C associated to applied pressure of 19500.

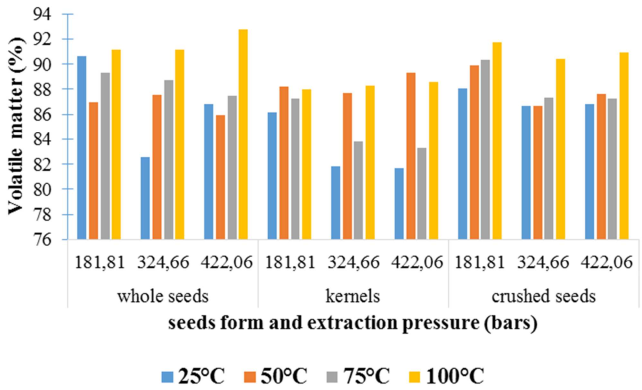

Figure 1. Cake volatile matter as a function of seed type, preheating temperature and extraction pressure.

In general, whatever the extraction pressure and preheating temperature, whole and crushed seeds registered the highest volatile matter compared to kernels. Indeed, the presence of husks would offer a greater amount of material compared to kernels. The cakes from the extraction of seeds preheated at 100°C associated to the extraction pressures of 15000 pounds and 19500 pounds obtained the higher volatile matter content. At these conditions, lipid expansion combined with compressive forces would occur by causing the breakage of oleic cell walls. This combined action would result in an increase in the amount of volatile matter

3.2.2. Residual Lipid Content

The highest residual lipid level (figure 2) independendly of the preheating temperature, the applied pressure and the seed form, is obtained with the cake from the kernels extraction preheated at 75°C associated to the lowest extraction pressure (181,81 bars). The lowest residual lipid was obtained with cake from non-preheated crushed seeds extraction (25°C) at the highest extraction pressure 19500 pounds.

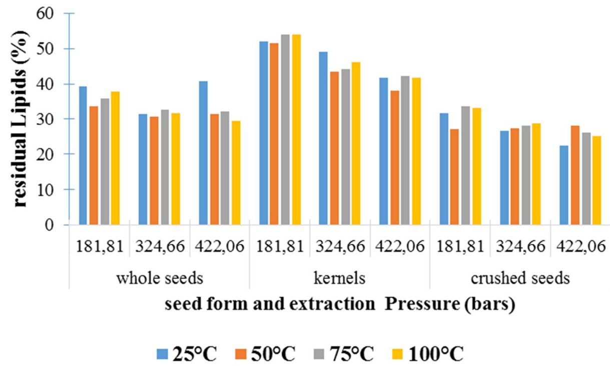

Figure 2. Residual lipids content as a function of Seed type, preheating temperature and extraction pressure.

Whatever the preheating temperature and the extraction pressure, the cake from kernels extraction obtained the highest residual lipid content. In fact, at equal weight, the oil content in Kernels is higher than that of whole seeds due to the presence of husks.

3.2.3. Cellulose Content

Regardless of the preheating temperature, extraction pressure, and seed type, the significantly highest cellulose content (figure 3) was obtained with the cake from the whole seed extraction preheated at 75°C associated at the applied pressure of 19500 pounds while the lowest was registered with the preheated kernels at 75°C associated to the lowest applied pressure. In general, whatever the applied pressure and preheating temperature, whole and crushed seeds had the highest cellulose content compared to kernels.

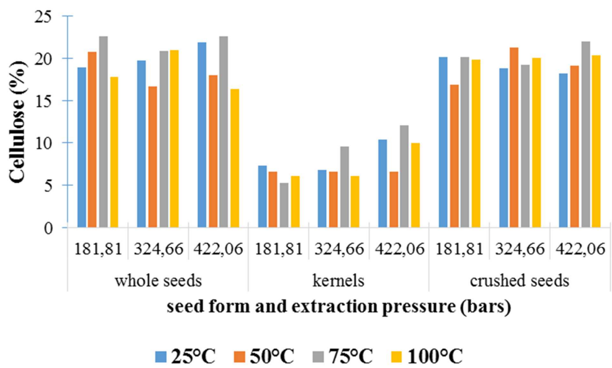

Figure 3. Cellulose content as a function of seed form, preheating temperature and extraction pressure.

No significant difference (p>0.05) was observed between the preheating temperatures. The higher the applied pressure the higher the cellulose content. These observation may due first of all to the presence of husks and secondly to reduction oil content which contribute to improve the cellulose content.

3.3. Biochar Yield as a Function of Seed Form, Preheating Temperature and Applied Pressure

The highest biochar yield (P <0.05) (table 2), regardless of preheating temperature, applied pressure and seed form, was obtained with cake from crushed seeds preheated at 100°C associated to the applied extraction pressure of 19500, the lowest value being observed in cake from non-preheated kernels extraction at the applied extraction pressure of 15000 pounds. Whole and crushed seeds with a high lignin content should have reduced mass loss. Indeed, during pyrolysis at low temperature, cellulose and hemicellulose degrade between 200 and 350°C while the decomposition of lignin is at 450°C [17-18-19]. For our pyrolysis experiments, at fixed temperature of 300°C, the mass conversion was higher with cake from kernels.

Table 2. *Effect of seed type, preheating temperature and applied extraction pressure on biochar yield.*

Seedforms	Applied pressure	Preheating temperature			
	(pounds)	25°C	50°C	75°C	100°C
Whole seeds	8400	68,02 ± 1,04aC*	53,68 ± 0,52aB*	52,55 ± 1,21aB*	63,67 ± 3,10aA*
	15000	57,83 ± 3,00aC**	50,78 ± 0,78aB**	54,86 ±1,78aB**	68,37 ± 0,58aA**
	19500	52,73 ± 2,19aC*	50,99 ± 1,00aB*	52,43 ± 0,56aB*	71,26 ± 2,88aA*
kernels	8400	47,40 ± 3,73cC*	48,78 ± 2,90cC*	53,80 ± 2,06cB*	57,63 ± 1,09cA*
	15000	41,75 ± 1,59cC**	53,96 ± 2,91cB**	43,06 ± 1,14cB**	63,91 ± 0,52cA**
	19500	45,41 ± 7,17cC*	61,52 ± 1,74cB*	45,40 ± 2,66cC*	67,59 ± 1,04cA*
Crushed seeds	8400	46,08 ± 0,72bC*	53,54 ± 1,93bB*	64,01 ± 1,57bA*	64,34 ± 1,91bA*
	15000	43,84 ± 0,71bC**	50,50 ± 0,17bB**	51,09 ± 0,98bB**	68,17 ± 2,90bA**
	19500	45,07 ± 0,64bC*	54,63 ± 0,96bB*	54,35 ± 1,46bB*	75,76 ± 1,53bA*

a, b, c: means with the same letter on the same column are not significantly different (p>0,05)
A, B, C: means with the same letter on the same line are not significantly different (p>0,05)
*: means with the same number of star in the same column are not significantly different (p>0,05)

Whatever the seed form or type, the highest biochar yield was observed with the preheating temperature/extraction pressure couple of 100°C-19500 pounds. Although very few authors highlight this aspect as a function of the couple. In fact, this couple would produce acceptable residual lipid content favoring low oxygenation of the carbon chains for a rich and efficient carbonization. This could also justify the low biochar yield from pyrolysis of kernel cake containing a higher residual lipid content compared to whole and crushed seeds.

3.4. Fixed Carbon as a Function of Seed Form, Preheating Temperature and Applied Extraction Pressure

The highest fixed carbon (p <0.05) under all considered of preheating temperature, extraction pressure and seed form, was obtained with biochar from non-preheated kernels associated to the extraction pressure of 15000 pounds, the lowest value being observed in biochar from whole seeds preheated at 100°C associated to the extraction pressure of 19500 pounds. No significant difference was found between the whole and crushed seeds. Carbonization also highlights the fixed carbon or pure carbon content of charcoal, which is of great importance in the field of metallurgy [20].

Our results show that fixed carbon contents are between 10 and 18% which are similar to those found in the literature [21-22-16]. A negative correlation was found between fixed carbon and biochar yield. Indeed, a high biochar yield induced a reduced mass loss with high volatile matter content. On the other hand, the determination of the fixed carbon is done by biochar combustion at high temperature, causing more volatile matter losses and carbon stabilization in the material.

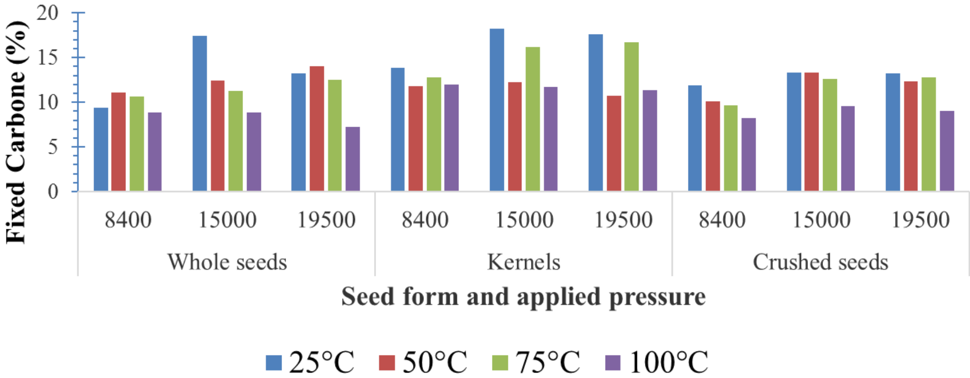

Figure 4. *Fixed carbone as a function of seed form, preheating temperature and applied pressure.*

3.5. Biochar Minerals Matter as a Function of Cake Type

3.5.1. Potassium

Independently of the seed form, the extraction pressure and the preheating temperature, the highest potassium percentage was obtained with biochar from kernels seed cake associated to preheating temperature/extraction pressure couple of 25°C-19500 pounds.

Table 3. *Effect of Seed type, extraction pressure and preheating temperature on the potassium content.*

Seedform	Applied pressure	Preheating temperature			
	(pounds)	25°C	50°C	75°C	100°C
Whole seeds	8400	1,89	2,08	2,08	1,81
	15000	2,62	2,78	2,02	1,50
	19500	2,87	2,84	2,21	1,64
Kernels	8400	2,47	1,92	2,13	0,01
	15000	2,69	2,02	2,75	2,00
	19500	3,24	1,62	2,91	1,41
Crushed seeds	8400	2,21	1,86	1,32	1,43
	15000	0,62	2,78	2,13	1,50
	19500	1,64	2,05	2,62	1,86

The lowest value being observed in biochar from kernel again associated to the couple 100°C-8400 pounds. The potassium levels are between 1.60 and 3.24%. These values are higher than those found (1.00 and 58,00 g/kg) by [23].

3.5.2. Calcium

The highest calcium value, independently of preheating temperature, extraction pressure and seed type, was obtained with biochar from kernels associated to the preheating temperature/extraction pressure couple of 25°C-19500 pounds the lowest value being observed in biochar from kernels associated to the couple of 100°C-8400 pounds.

Table 4. *Effect of Seed type, extraction pressure and preheating temperature on the Calcium content.*

Seedform	Applied pressure	Preheating temperature			
	(pounds)	25°C	50°C	75°C	100°C
Whole seeds	8400	0,34	0,28	0,34	0,32
	15000	0,44	0,30	0,30	0,28
	19500	0,44	0,40	0,38	0,32
Kernels	8400	0,39	0,42	0,40	0,08
	15000	0,50	0,34	0,40	0,34
	19500	0,54	0,32	0,44	0,20
Crushed seeds	8400	0,32	0,40	0,26	0,26
	15000	0,16	0,38	0,34	0,24
	19500	0,14	0,28	0,32	0,26

Our results on the calcium levels (0.08 and 0.44%) are lower compared to those founds (1 and 2.00%) by [24] with biochar derived from wood trunks. This difference may be simply due either to the higher lignin content in wood or to the lower ash content in jatropha seeds.

3.5.3. Magnesium

Independently of the seed form or type, the extraction pressure and the preheating temperature, the highest magesium percentage was obtained with biochar from kernels seeds associated to preheating temperature/extraction pressure couple of 25°C-15000 pounds. The lowest value is observed in biochar from kernel preheated at 100°C associated to the highest extraction pressure (19500 pounds).

Table 5. *Effect of Seed type, extraction pressure and preheating temperature on the Magnesium content.*

Seedform	Applied pressure	Preheating temperature			
	(pounds)	25°C	50°C	75°C	100°C
Whole seeds	8400	0,056	0,056	0,044	0,044
	15000	0,068	0,092	0,068	0,056
	19500	0,056	0,068	0,068	0,056
Kernels	8400	0,075	0,068	0,056	0,046
	15000	0,08	0,068	0,068	0,056
	19500	0,068	0,056	0,044	0,007
Crushed seeds	8400	0,032	0,056	0,044	0,044
	15000	0,044	0,068	0,019	0,032
	19500	0,032	0,068	0,068	0,044

3.5.4. Phosphorus

The highest phosphorus value independently of preheating temperature, extraction pressure and seed form, was obtained with biochar from kernels associated to the preheating temperature/extraction pressure couple of 100°C-15000 pounds, and the lowest value in the biochar from kernels once again associated to the couple of 100°C-8400 pounds. The phosphorus content between 0.30 and 1.00% is higher compared to those (0.20 to 0.73 g/kg) obtained by [23].

Table 6. *Effect of Seed type, extraction pressure and preheating temperature on the Phosphorus percentage.*

Seedfor m	Applied pressure	Preheating temperature			
	(pounds)	25°C	50°C	75°C	100°C
Whole seeds	8400	0,60	0,45	0,64	0,72
	15000	0,58	0,62	0,56	0,72
	19500	0,66	0,68	0,62	0,66
Kernels	8400	0,72	0,72	0,72	0,09
	15000	0,72	0,72	0,79	1,02
	19500	0,75	0,74	0,62	0,58
Crushed seed	8400	0,68	0,58	0,49	0,37
	15000	0,41	0,66	0,60	0,43
	19500	0,53	0,56	0,60	0,58

3.5.5. pH

The characteristics of the produced jatropha biochar identified previously by the inorganic nutrients are useful in fertilization and plant growth. In addition whatever the preheating temperature, the extraction pressure and the type of the seeds, our results show a biochar with a pH between 7.5 and 10, highly alkaline. These values are very similar to pH 7 and 11 found by [25] in biochar from Pongamia glabra.

4. Conclusion

Oil extraction conditions and the seed form in particular, have a significant influence on volatile matter, residual lipids and cellulose content. No significant difference was observed between dry matter content. Seed form significantly affected the cake yield and biochar yield. Indeed, they were higher with crushed seeds. The type of cake also influenced the fixed Carbone level. Thus the highest value is recorded in biochar from jatropha seeds at the preheating temperature/extraction pressure couple of 25°C-15000 pounds. A negative correlation exists between the biochar yield and the fixed Carbon level. Although the type of cake did not significantly affect the biochar minerals content, the highest value for K, P, Mg and Ca was observed in the biochar from kernel cake. In general, the biochar from the jatropha cakes pyrolysis can be of great use in acid soil fertilization.

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