

Research/Technical Note

Effect of Oil Extraction Conditions on the Anaerobic Fermentation of *Jatropha curcas* Seed Cakes

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To cite this article:

Sogang Segning Harry Bertholt, Nsah-ko Tchoumboue, Djousse Kanouo Boris Merlain, Tangka Julius Kewir. Effect of Oil Extraction Conditions on the Anaerobic Fermentation of *Jatropha curcas* Seed Cakes. *Science Journal of Energy Engineering*. Vol. 9, No. 1, 2021, pp. 1-7. doi: 10.11648/j.sjee.20210901.11

Received: November 16, 2020; **Accepted:** December 14, 2020; **Published:** January 12, 2021

Abstract: The production of biogas is a healthy way to convert biomass like seed cakes into energy. But before that, oil seeds like *jatropha curcas* are treated in some conditions in order to get oil first, and cakes after. And different levels of those conditions that are seed type, preheat temperature and extraction pressure can give different results on biogas production using those cakes. That is why in this study, biogas characteristics of cake, from three types of *jatropha* seeds (whole, kernels and crushed) preheated at four levels of temperature (25°C, 50°C, 75°C and 100°C) and submitted at three extraction pressures (181.81 bar, 324.66 bar and 422.06 bar), were assessed. It consisted in mixing 5 ml solid mixture (2.5 ml of cake sample and 2.5 ml cow dung) to the buffer solution (NH₄Cl, 100; NaCl, 10; MgCl 2 · 6H₂O, 10; CaCl₂·2H₂O, 5) at 1:1 ratio. The mixture was introduced in a reactor of 16 ml volume and then incubated in an electric oven at 37°C for 60 days. The measure of gas volume of each sample was done using the adapted method of displaced liquid. The gas calorific value estimation of each sample was theoretically done, using Buswell equation. And, the gas energy was obtained for each sample, using its gas volume and calorific value. The main results were as follows. The highest volume of biogas (8 ml / g) and highest energy (2485.85 J) were registered with whole seed cake at preheated temperature/extraction pressure couple of 100°C-324.66 bar. The highest calorific value (701.43 KJ/mol) was obtained with the whole seed cake at preheated temperature/extraction/ pressure couple of 100°C-422.06 bars. The main conclusion that can be made is that, there is an effect of oil extraction conditions on the anaerobic fermentation of *jatropha curcas* seed cakes.

Keywords: Biogas, Oil Seeds, *Jatropha*, Seed Cake, Volume, Heating Value, Energy

1. Introduction

Energy is essential for the social well-being and economic development of any country [8]. At the global level, energy is designated as a priority for the 2015-2024 decade and is one of the sub-2criteria of the Sustainable Development Goals [18]. These goals are more relevant to developing countries where energy needs are still high and growing. Indeed, more than 3 billion people, mostly in Asia and sub-Saharan Africa, still do

not have access to energy [17]. Without additional efforts, it is estimated that by 2030, access to electricity and clean cooking technologies will remain 42% and 56% respectively [12]. In Cameroon, which represents the second largest African hydroelectric potential after the Democratic Republic of Congo [21], the rate of electricity access and cooking gas is of 60.1 and 15.3% respectively [15]. So far, oil, natural gas and coal are the world's leading sources of energy. Despite the efforts made in Africa to reduce the energy deficit,

potentialities are still under exploited. These energy sources have some drawbacks such as greenhouse gas emissions and their exhaustible aspect not to mention the permanent increase in prices [16]. It therefore makes sense to look other alternatives such as biomass due to its availability. But the methods of using this biomass are still archaic, for example in Cameroon, 99% of the rural population still uses wood as fuel [20]. This causes many respiratory and eye diseases and also global warming. These problems can be avoided or considerably limited by opting for the valorization of biomass by the biogas process. In this respect, non-edible oilseeds have a triple interest in firstly the production of biofuel, then the energy recovery of the extraction residues by the biogas process, and finally that these uses have little impact on the environment and food security. Although work has been done on the production of biogas from jatropha cakes [6], there is still a lack of scientific and technical knowledge on oil extraction processes that would optimize biogas yields from residues; however, recent works has shown that oilseed oil extraction processes influence the characteristics of extraction residues [6, 19]. The purpose of this study was to study the effect of the extraction biofuel oil conditions of the *Jatropha curcas* seeds on the biogas characteristics.

2. Materials and Methods

2.1. Provenance and Processing of Samples

Jatropha seeds were collected in Jakiri, North West Cameroon, and were used to extract oil for use as a biofuel. This oil extraction was done on three states of *Jatropha* seed which are complete seeds, kernels and crushed seeds, all presented in Figure 1.



Figure 1. The three states of *Jatropha* seeds used. (a) whole seeds, (b) kernel and (c) crushed seeds.

The seeds in each of the three states were before extraction, subjected to four preheating temperatures which are 25°C, 50°C, 75°C and 100°C. The oil was then extracted by a mechanical press (hydraulic cylinder) at pressures of 181.81 bar, 324.66 bar and 422.06 bar, according to the experimental design illustrated in figure 2. Each of these treatments has been repeated three times.

2.2. Data Collection

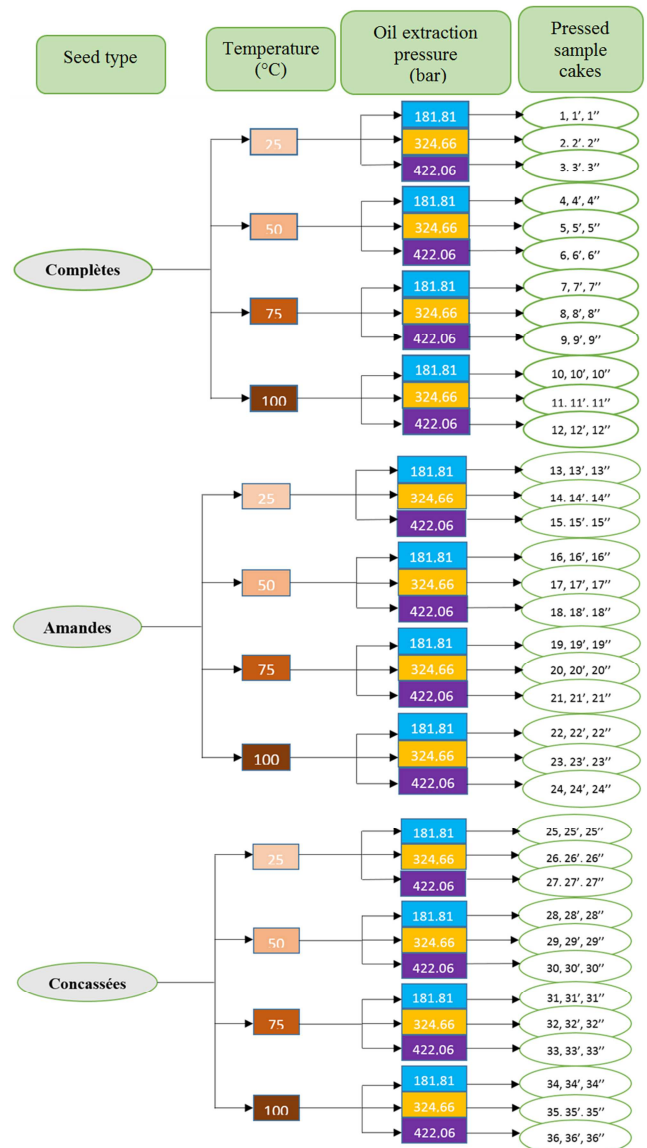
2.2.1. Characterization of Cakes

It was carried out by the Association of Official Analytical Chemists (AOAC) method [3], and consisted of determining percentages of volatile matter, solids, residual lipids and cellulose. These four parameters are the main ones likely to undergo changes under the effect of the oil extraction conditions of the seeds [10].

2.2.2. Establishment of Experimental Units

The size of the samples was first reduced in a Moulinex to facilitate degradation, before sampling 2.5 ml of each in its bottle. The already fermented cow dung (no risk of this producing extra biogas) of 2.5 ml per bottle from a biodigester was used as an inoculum for microbial biomass input responsible for methane production. For the control of pH neutrality during fermentation, a 5 ml buffer solution was used [2]. The chemical formula and concentration values in g / l of distilled water used to obtain the buffer solution are as follows [2]: NH_4Cl , 100; NaCl , 10; $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$, 10; $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$, 5.

The blends were therefore carried out and placed in clean labeled glass bottles of 16 ml capacity used as reactors, then sealed under ambient air conditions and put into production at 37°C (suitable for mesophilic bacteria) for 60 days [1], as shown in Figure 3.



“complète” = whole; “amande” = kernel; “concassée” = crushed

Figure 2. Experimental design for oil extraction from *Jatropha* seeds.

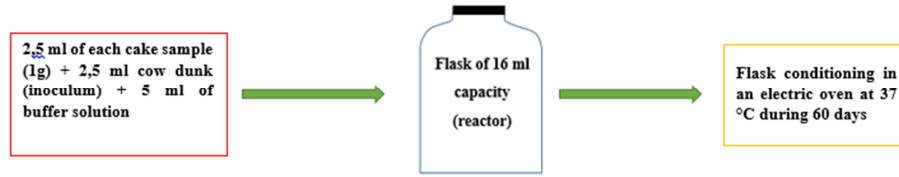


Figure 3. Experimental device for setting up experimental units.

2.3. Measurement of Characteristic Parameters of Biogas Produced

2.3.1. Measuring the Volume of Biogas from Each Flask

The volume of biogas produced by each bottle was measured by the adapted method of displaced liquid [13] under the conditions of ambient temperature (25°C) and atmospheric pressure (1 atm). The liquid used here is drinking water free of any particles. Figure 4 illustrates the principle of this method in a, and its realization in b [5].

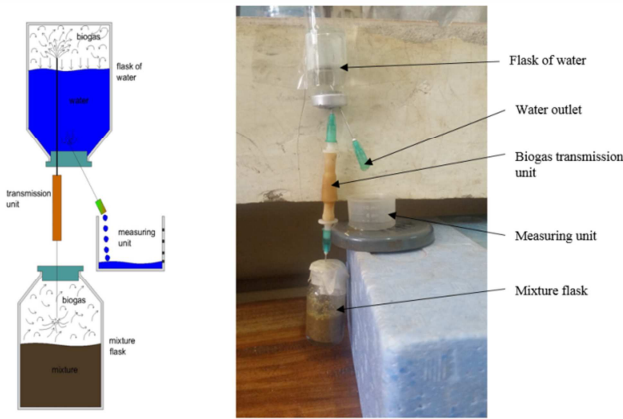
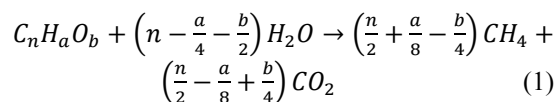


Figure 4. The adapted method of displaced liquid.

2.3.2. Calculation of the Calorific value of Biogas from Each Flask

The calorific value is closely related to the methane content of the biogas produced. To ensure the presence of methane in the flasks, the control flasks of each sample were tested on combustion and a very fast flame was observed (due to the very small quantity of the samples) which testifies to the presence of methane. But as for the percentage of methane in the biogas produced, for lack of adequate measuring equipment like gas chromatography and many others, it has been estimated theoretically thanks to the Buswell equation [7] illustrated by Equation 1 below, where the organic substrate (C_nH_aO_b) in the presence of water (H₂O) ferments anaerobically to produce proportions of methane (CH₄) and CO₂.



From which was easily derived the formula of the rate of methane here in equation 2, using the indices of proportion present in front of each element.

$$\%CH_4 = \left(\frac{1}{2} + \frac{a}{8n} - \frac{b}{4n}\right) \times 100 \quad (2)$$

But to be able to apply this formula, it is needed to know the empirical formula (C_nH_aO_b) of each sample. For this, we proceeded by the method of quantitative analysis by complete combustion of each of the samples in an electric furnace in the laboratory. Knowing that the methane has a lower heating value of 804.3 KJ / mol at 25°C, the heating value of the biogas produced by each sample was thus determined by the following formula 3 [7]:

$$PC_{biogas} = \%CH_4 \times PCI_{CH_4} \quad (3)$$

PC_{biogas} is the heating value of biogas in KJ / mol

% CH₄ is the percentage of biogas methane in%

PCI_{CH₄} is the lower heating value of methane in KJ / mol

2.3.3. Calculation of the Biogas Heat Energy of Each Flask

The sample with the best volume yield is not necessarily the one with the highest calorific value, so we will not be able to select the best sample at the end of the production. For this reason the calorific energy parameter is used to serve as a combination of volume and heat value. As the heat energy of a gas is defined as the calorific value per unit volume, it has been calculated for each sample by the following formula 4:

$$EC_{biogas} = PC_{biogas} \times V_{biogas} \quad (4)$$

EC_{biogas} is the calorific energy of biogas in J

PC_{biogas} is the calorific value of biogas in KJ / mol

V_{biogas} is the volume of biogas in mol

2.4. Statistical Analysis of the Data

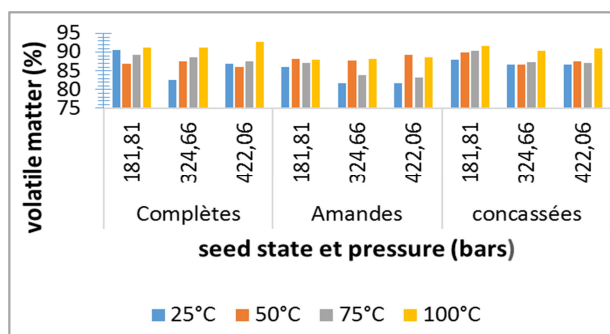
All data were subjected to an analysis of variance (ANOVA) and the results were compared using the Fischer test. The separation and classification of averages were done by Duncan's test. Correlations by the Pearson test were made between the values of the biogas parameters and the characteristics of cakes to see if there is a link between them. The software used for all these analyzes was SPSS 23, and the probability threshold was 5%.

3. Results and Discussion

3.1. Characteristics of the Samples

3.1.1. Volatile Matter (VM)

The volatile material levels obtained from each sample of cake are illustrated in Figure 5 below.



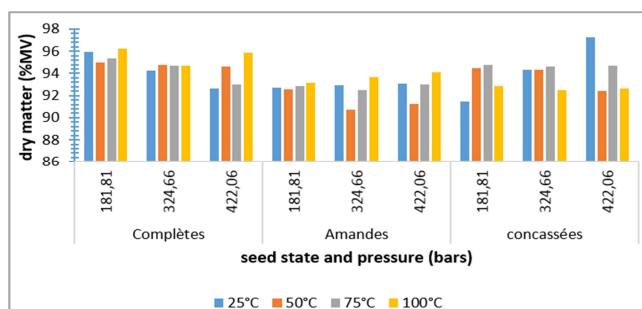
“complète” = whole; “amande” = kernel; “concassée” = crushed

Figure 5. Percentage of volatile matter per sample.

This shows that regardless of the preheat temperature, extraction pressure, and seed condition, the significantly highest (92.77%) and the lowest (81.71%) volatile matter, were respectively obtained in the cake from the pressing of the whole grain (100°C and 422.06 bars) and that from the pressing of the almond seed (25°C and 422.06 bars). With an average of 87.24%, close to 84.88% obtained by [19]. After statistical analysis of the data, it was found that crushed seed cakes and whole seed cakes contained on average more volatile matter than the kernels. This could be due to the fact that the crushed and whole seeds contain husk which are additional material; similarly, these husks would have increased the resistance to extraction, which would have allowed these seeds to keep a large part of their oil, thus increasing the rate of volatile matter.

3.1.2. Dry Matter (DM)

The DM obtained from each sample of cake is illustrated in Figure 6 below. It appears that regardless of preheat temperature, extraction pressure and seed state, the significantly highest (92.28%) and lowest (90.72%) DM levels, was respectively obtained by the cake from the pressing of the crushed seed (25°C and 422.06 bars) and that from the pressing of the kernel seed (50°C and 324.66 bar). These results give an average of 91.5% which is closed to 93% obtained by [9].



“complète” = whole; “amande” = kernel; “concassée” = crushed

Figure 6. Percentage of dry matter per sample.

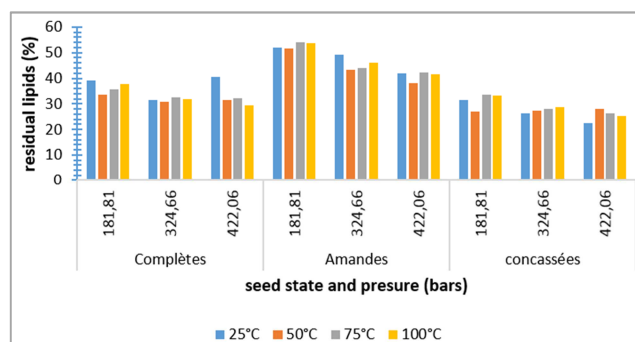
Statistical analyzes of the data revealed that whole seed cakes followed by crushed seed cakes has an average higher DM content than kernels cakes. This may have been due to the fact that for the same mass of these seeds, the whole and the

crushed seeds have shells which possess less water than the kernel portion of the seed; unlike kernels that have no husk. This explains that the level of DM is higher in the cakes of whole and crushed seeds compared to kernels cakes.

3.1.3. Residual Lipids

The residual lipid levels obtained from each sample of cake are illustrated in Figure 7 below.

It shows that regardless of the preheating temperature, the extraction pressure and the state of the seed, the significantly highest (53.94%) and lowest (22.47%) residual lipid content, were respectively obtained of cakes from the pressing of the kernel seeds (75°C and 181.81 bar) and that from the pressing of the crushed seeds (25°C and 422.06 bars). Some [11] obtained about 20% of residual lipids after extraction of the kernels seeds; [19] by increasing the extraction pressure have gotten a small margin of 6.40% (with shells) at 6.32% (kernels).



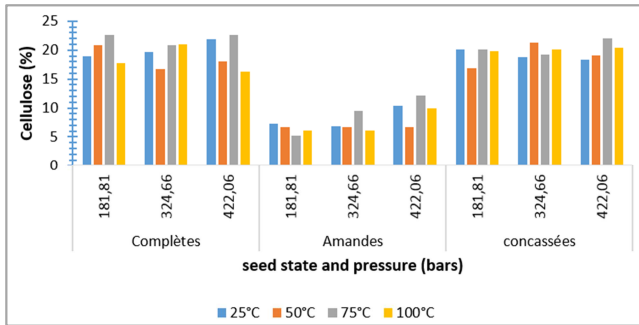
“complète” = whole; “amande” = kernel; “concassée” = crushed

Figure 7. Percentage of residual lipids per sample.

These results are different because the extraction pressures are different too. It follows from statistical analysis of data that kernel seed cakes had on average the highest percentage of residual lipids compared to those of whole and crushed seeds. This would have been due to the fact that initially for the extraction, the same mass for these three states of the seeds was taken. The oil of the seed of *Jatropha* being essentially contained in the kernel part, then the mass of the kernel initially had more oil than this same one mass for the crushed and whole seeds, because of the presence of the husk which reduces their quantity of kernel containing oil. When these three seed states are therefore subjected to the same extraction pressure, kernel residues will always retain more residual lipids than those of crushed and whole as at the beginning.

3.1.4. Cellulose

The cellulose levels obtained from each sample of cakes are illustrated in Figure 8 below. These results indicate that independently of preheating temperature, extraction pressure and seed state, the significantly highest (22.64%) and lowest (5.25%) cellulose levels were respectively obtained in the cake from the pressing of the whole seeds (75°C and 422.06 bars) and that from the pressing of the kernel seeds (75°C, 181.81 bar). Results are similar to those of [19] which obtained for cakes with husks 20.3%, and 6.43% for kernels.



“complète” = whole; “amande” = kernel; “concassée” = crushed

Figure 8. Percentage of cellulose per sample.

As a result of the statistical analysis of the data, it was noted that cakes from crushed and whole seeds had on average a higher level of cellulose than kernel seed residues. The reason for this difference is the presence of the husks in the cakes of whole and crushed seeds compared to the kernel seeds which do not have any husks, when it is in husks that most of the cellulose is found for *Jatropha* seeds, and many other seeds.

3.2. Biogas Volumes of Samples

Following the measurement of the biogas volumes in each flask, biogas volume values were obtained and illustrated in Figure 9. It was obtained independently of influencing factors, a max volume of 8 ml and a minimum of 1, 25 ml, respectively from cakes of the whole seeds (100°C and 324.66 bar) and that of the whole seeds (75°C and 181.81 bar). Moreover, there were significant correlations of 0.42 and -0.37 between the volume of biogas and respectively the volatile matter and the residual lipids.

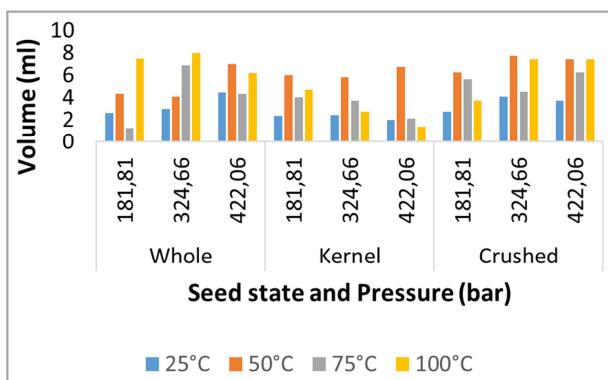


Figure 9. Volume of biogas per sample.

This means that more a sample contains volatile matter, more biogas it produces in terms of volume because volatile matter is a biodegradable part of the material that produces biogas [4]. And the more a sample contains residual lipids, the less biogas it produces; this is because lipids produce volatile fatty acids which at a certain level acidify the fermentation medium, thus inhibiting the activity of the microorganisms responsible for the production of methane, hence the drop in yield [22].

3.2.1. Effect of Seed Form

Statistical analyzes revealed that the group of crushed seeds produced on average the best yield in volume (5.57 ml), followed by that of whole seeds (4.97 ml) and finally that of kernel seeds (3.67 ml). However, the cakes of the crushed seeds contain more volatile matter, followed by those of the whole seeds and kernel seeds. Knowing that volatile matter is the degradable part that contributes to biogas production, it is clear that the more biogas there is, the greater the volume of biogas. In addition, kernels contain more residual lipids followed by crushed and complete seeds.

3.2.2. Effect of Preheating Temperature

Following the statistical analysis of data, seed residues preheated to 50°C gave the best average yield by volume of biogas (6.15 ml), followed by those of seeds preheated to 100°C (5.45 ml), 75°C (4.30 ml) and 25°C (3.04 ml). And cakes preheated to 100°C and 50°C contain on average more volatile matter and less residual lipids than those preheated at 25°C and 75°C. However, since volatile matter favors the production of biogas and the residual lipids inhibit it from a certain threshold, it is obvious that samples preheated at 100°C and 50°C contain more volatile matter and less residual lipids produce more biogas than those preheated at 25°C and 75°C that contain less volatile matter and more residual lipids.

3.2.3. Effect of Extraction Pressure

Statistical analysis of the data revealed that the cakes pressed at 324.66 bar gave the best average volume yield of 5.03 ml, followed by those pressed at 422.06 bar (4.92 ml) and 181.81 bar (4.26 ml). However, the cakes pressed at 181.81 bars have average levels of MV and residual lipids higher than those of the cakes pressed at pressures 324.66 bars and 422.06 bars. The residual lipids having an inhibitory effect at a certain threshold, it is quite normal that the samples pressed at 181.81 bars which possess more, produce less biogas than those pressed at 324.66 bars and 422.06 bars which have them less. It is nevertheless noted that the cake pressed at 181.81 bars possess more volatile matter than the two others, but still produce less biogas than the latter; this is justified because the surplus volatile matter they have are the residual lipids because they have it more, and these lipids have rather an inhibitory effect. So it is normal that these cakes pressed at 181.81 bar produce less biogas than those pressed at 324.66 bar and 422.06 bar.

3.3. Calorific Value of the Biogas

As a result of the calculations, a methane range of 84% to 87% was obtained, giving calorific values of biogas in each flask, obtained and illustrated in Figure 10.

It shows that independently of the influence factors, the maximum calorific value (701.43 KJ / mol) was obtained from the whole seed cakes (100°C, and 422.06 bar), and the minimum (660.65 KJ / mol) was obtained from the residues of the kernel seeds (25°C, 422.06 bar).

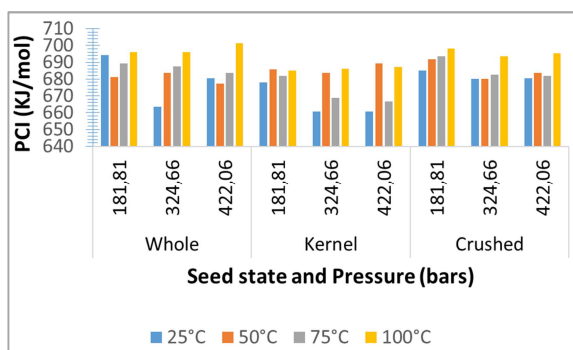


Figure 10. Calorific power values of biogas per sample.

Among other things, there were significant correlations of 0.99 and -0.28 between calorific value and, respectively, volatile matter level and residual lipids. This means that the more a sample contains volatile matter, the more it produces biogas in terms of calorific value because volatile matter is a biodegradable part of the material that produces biogas [7]. And the more a sample contains residual lipids, the less biogas it produces; this is because lipids produce volatile fatty acids which for a certain threshold acidify the fermentation medium, thereby inhibiting the activity of the microorganisms responsible for the production of methane, hence the drop in the heating capacity yield [14].

3.3.1. Influence of the State of the Seed

The statistical analyzes revealed that cakes from crushed seeds and whole seeds groups produced on average the best calorific yields (respectively 687.16 KJ / mol and 686.21 KJ / mol), compared to that of the kernel seed cakes. (677.80 KJ / mol). And it is known that the cakes of the crushed seeds and whole seeds groups contain on average more volatile matter and less residual lipids than those of the group of kernel seed cakes. So in view of the correlations, it is justified that these first two groups produce a biogas with a higher calorific value compared to that of kernels.

3.3.2. Influence of the Preheating Temperature

Following the statistical analysis of the data, the residues of seeds preheated to 100°C gave the best average yield of calorific value of biogas (693.12 KJ / mol), followed by those of seeds preheated to 50°C (684.03 KJ / mol), 75°C (681.80 KJ / mol) and 25°C (675.94 KJ / mol). Recalling that cakes preheated to 100°C and 50°C contain more volatile matter and less residual lipids than those preheated to 75°C and 25°C, it is normal that they produce a biogas with calorific powers higher than those of the last two.

3.3.3. Influence of the Extraction Pressure

Statistical analysis of the data revealed that the oilcakes pressed at the pressure of 181.81 bar gave the best average yield of calorific value (688.31 KJ / mol), followed by those pressed at 422.06 bar (682.35 KJ / mol) and 324.66 bar (680.51 KJ / mol). This is normal because the samples pressed at 181.81 bar contain more volatile matter on average compared to 324.66 bars and 422.06 bars. However, cakes pressed at 181.81 bar have a higher residual lipids content than

those pressed at 324.66 bar and 422.06 bar, while producing a better calorific value than the last two; which corroborates the assertion of [7] that the more lipids the substrate contains, the higher the heating value of the biogas resulting from its fermentation at a certain threshold.

3.4. Calorific Energy of Biogas Samples

The deduction of the calorific energies made it possible to obtain values which have been illustrated in figure 11.

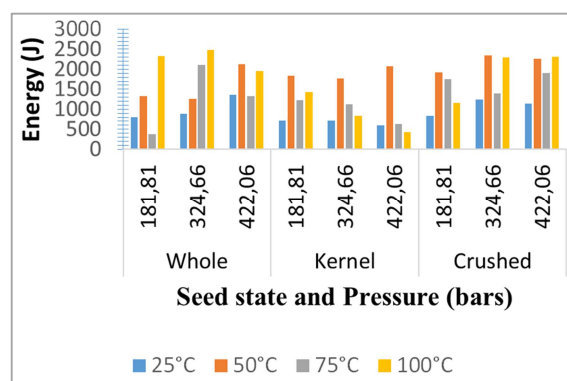


Figure 11. Biogas heat energy per sample.

And whatever the influence factor, the highest value significantly in terms of heat energy was 2485.85 J. obtained from the residue of the whole seed (100°C, 324.66 bar), and the lowest of 384.69 J obtained from the cake of the whole seed (75°C, 181.81 bar). There were also significant correlations of 1 and 0.44 between the heat energy and the volume and the calorific power value of biogas, respectively. This shows that the heat energy depends more on the volume of biogas than on its calorific value. This can be explained by the fact that there are very large differences or variability between the biogas volume values of the samples (84%) compared to those of the calorific power values (6%). This means that the influence of the seed state, preheat temperature and extraction pressure on the heat energy is identical to their influence on the biogas volume.

4. Conclusions

Jatropha seed oil extraction conditions such as seed state, preheating temperature and extraction pressure have a significant influence on the physicochemical characteristics of their residues. Consequently, these conditions also influence the production parameters such as the volume, calorific value and heat energy of biogas based on these residues. The fact is that with these varieties of treatments, we have obtained a volume range of 1.25 ml to 8 ml, a calorific value range of 660.65 KJ/mol to 701.43 KJ/mol, and a heat energy range of 384.69 J to 2485.85 J.

5. Recommendations for Follow-up

Recommendations for future works are as follows:

Assess the percentage ranges where lipids will become an

inhibitory factor for biogas production; analyze gas samples of this study with a gas chromatography technology in order to confirm results obtained theoretically; also check about the influence of other factors that have not been taken in consideration in this study, on the biogas production parameters.

Acknowledgements

This work was supported by Renewable Energies Laboratory of the University of Dschang, with the collaboration of biochemistry laboratory, organic chemistry laboratory and soil sciences laboratory of the same University.

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