



Research article

The effects of moisture content, fiber length and compaction time on African oil palm empty fruit bunches briquette quality parameters

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ARTICLE INFO

Keywords:

Mechanical engineering
 Food science
 Agricultural science
 Empty fruit bunches (EFB)
 Moisture content
 Fiber length
 Compaction time
 Briquettes density
 Durability index
 Compression strength

ABSTRACT

In this paper a study on the process of densification of oil palm empty fruit bunches (OPEFB) is presented. An empirical-statistical model that allows the evaluation of densification process is obtained through an experimental factorial design. The main purpose of the experimental arrangement is to find the appropriate reference values for the experimental factors - moisture content, fiber length and compaction time-with which optimal performance responses of briquettes are achieved. Statistical models are obtained that explain in an acceptable way the influence of independent experimental factors on the mechanical properties of briquettes, such as briquettes density, durability index and compressive strength. It is possible to conclude that briquettes manufactured with the appropriate reference values, moisture content of 8% w.b., fiber length of 73.6 mm and a compaction time of 26.6 s respectively, meet mechanical and thermal requirements that are required in the most representative standards for biomass briquettes for energy purposes. Results obtained in the current investigation can be used as reference for the design of an industrial pilot plant destined to the densification of EFB of African oil palm.

1. Introduction

Biomass is an energy source from renewable organic matter of animal or plant origin, or product of artificial or natural transformation (Kirkels, 2016). Particularly, residual biomass has a high exploitation potential due to its low cost, high availability and sustainability (Chew et al., 2019).

On the other hand, greenhouse gases reduction is required as a measure to reduce the effects of climate change. Studies show that the use of biomass to produce fuels, like substitutes for petroleum and coal derived fuels; they would reduce the production of CO₂ (Kajikawa and Takeda, 2008). The use of biomass from residues of African oil palm (*Elaeis Guineensis*) would reduce emissions from CO₂ from 17.4 Tg p/year to 12.6 Tg p/year and from 350 PJ of oil p/year to 230 PJ of oil p/year. That is, 72% and 67% of the reductions respectively, obtaining an approximate monetary benefit of 45 million euros p/year (Gustavsson et al., 2007).

In Colombia, according to the latest Fedepalma census 8682 Tn/h of African Oil Palm fruit are extracted in 43 plants installed in the country; of which, 1755 Tn/h are empty fruit bunches of African oil palm (hereafter called EFB) (Rivera-Méndez et al., 2017). At present, EFB is used as

fertilizer for plantations; being deposited between grooves of palm crops. However, EFB transport to the crops causes compaction of soils where the crops are located, resulting in a production decrease. However, that EFB could be used in the production of fuel at a competitive cost, in relation to petroleum-derived fuels, a characterization of its densification process is required. It has been identified that to prevent premature deterioration of EFB, its prior drying is required until a moisture content of less than 15% w.b. (Tang et al., 2016).

Regarding a possible standardization and regulations for manufacture and evaluation of quality of briquettes produced from EFB, the fact that Europe has the most complete regulations concerning manufacturing and North America concerning evaluation stands out. Among European standards are: DIN 51731 about pellet and briquette manufacturing standards, and DINEN 15270 about manufacture of high-quality pellets (Liu et al., 2016). ÖNORM M 7135 establishes requirements and specifications for the manufacture of wood pellets and briquettes implemented in Austria (Oberberger and Thek, 2004) and the French recommendation ITEBE, the standardization for the manufacture of briquettes (Ríos-Badrán et al., 2020), among others.

From the review, it is concluded that there is no comprehensive investigation to date to define the influence that parameters of EFB

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Table 1. Arrangement of the experimental design. Source: Authors.

Experimental factor	Symbol	Real value	Coded value
Moisture content, [%]	W8	8	-1
	W12	12	0
	W16	16	+1
Fiber length, [mm]	X	50	-1
	Y	85	0
	Z	120	+1
Compaction time, [s]	L	0	-1
	M	20	0
	H	40	+1

preparation have on the final mechanical and thermal properties of briquettes, such as fiber length and moisture content and the time to sustain compaction pressure. Therefore, the use and execution of an experimental design is proposed, whose results and analysis can be understood as main factors that define the biomass and the process, influence the response parameters. For this investigation, briquettes density, durability index and compressive strength are selected as response parameters.

The present study allows the characterization at an experimental level of the densification process of the EFB of African oil palm, in order to orient the results towards the large-scale implementation of the briquetting activity of this biomass for use as a biofuel.

2. Experimental methods

2.1. Experimental design

A complete three-level factorial design with three experimental factors was established. According to the review of the State of Art and a group of preliminary tests, ranges of values were established with the significant influence of experimental factors. Experimental factors and their levels for the final factorial design were selected based on preliminary experimental tests that allowed identifying factors with a greater effect and the most appropriate experimental levels. For this first exploratory phase, a strategy of varying one factor at a time was used, within a broader range than the experimental installation and physical conditions of the biomass allowed. Experimental factors were divided into two groups: essential and secondary. Among essential variables are moisture content whose measurement process was carried out in accordance with ASTM E 871 (Konrád et al., 2018); fiber length, whose measurement was based on a ruler previously graduated in guillotine; and compaction time, measured with the help of a stopwatch. On the

other hand, fixed values were established for secondary variables of compaction pressure and compaction temperature, with their values setting to 97 MPa and 90 °C respectively.

The selection of crops was made by area of origin in accordance with the division of crops of African Palm in the Colombian territory, which are found in: north, central and eastern zone respectively (Rivera-Méndez et al., 2017). Then, three experimental blocks are used, made up of biomass from three representative regions of the country about cultivation of African Oil Palm: Fundación, Sabana de Torres and Puerto Gaitán. Thus, results obtained from this study are representative for the whole country. Each sample is coded by region where the crop comes from: C1 (Fundación), C2 (Sabana de Torres), and C3 (Puerto Gaitán). Table 1 shows the arrangement of the experimental design. Each treatment has three replicas.

2.2. Samples preparation

The EFB sample selection process was carried out randomly after the process of fruit removal in each of the extraction plants. They were transported from the respective collection regions to the Laboratory in a period of less than 48 h to avoid fungi generation. Next, a moisture content measurement process was carried out, with an average value of 60% w.b.; and fibers were opened, and the controlled drying process was carried out in a biomass dryer. Fibers were cut according to values specified in the experimental design for fiber length. The oil palm empty fruit bunches fibers were precisely cut manually and using a graduated guillotine, while the moisture content of the biomass was established through the following procedure: first the biomass was dried to a low humidity, performing the measurement of its moisture content by using a moisture analyzer (OAHUS MB45) that was calibrated under the (ASAE) s 358.2 standard. This equipment uses a sample of 0.5 g, and by means of the variation of the weight of this sample during drying, the moisture content is calculated. Then, the percentage by mass of water necessary to reach experimental levels specified in the experiment design was added to each treatment; finally, biomass samples were placed in hermetic bags for 12 h to achieve the homogenization of the humidity prior to manufacture the briquettes.

Figure 1 shows different states through which raw material went through.

2.3. Procedure for obtaining briquettes

A hydraulic briquetting press is used to perform biomass densification. An image of the briquetting press used in the present investigation is shown in Figure 2. At present there is no specific equipment implemented



Figure 1. Raw material states from corozo to storage of cut fiber in airtight packages. Source: Authors.



Figure 2. Briquetting press developed for the current investigation. Source: Authors.

to compact residual biomass from African oil palm, with instrumentation for measuring the essential variables described above. The existing ones for compaction of biomass do not have temperature control, indispensable to achieve plasticization of briquettes (Fedepalma, 2016; Kaliyan et al., 2013). Matrices of the briquetting press were built with AISI/SAE H13, material with good performance at moderate temperatures. The diameter of the matrix (densification chamber) is 32 mm. Other main parts were made with AISI/SAE 4140 material to give greater mechanical robustness. The press has a hydraulic system with a capacity of 100 kN force and 50 cm stroke. In the present investigation, compression force to perform experimental runs was set at 78 kN. In addition, the hydraulic system at the top is connected to a kneecap to align and apply the axial force. The load cell is connected to the piston. The densification chamber is located inside an oven, where the temperature can be set between 50 °C and 200 °C. A suitable temperature enables the activation of lignin, which allows the union among biomass fibers. The briquette is removed from the top of the chamber. In addition, the press has a platform to allow safe and ergonomic work of the experimenter.

2.4. Tests to determine mechanical properties

Density test was based on the standard of the American Society of Agricultural and Biological Engineers (ASABE s269.4), which describes the procedure to calculate density (D_b) in briquettes and pellets (ASABE s269.4., 2007). Briquettes density measurement was obtained using the mass/volume ratio. The volume calculation was made with a calibrator (Vernier KANON with 0.05 mm accuracy), by measuring the dimensions of the briquettes. To measure the mass of briquettes, a precision scale (Ohaus PA224 - Pioneer Analytical Balance, 220 g \times 0.1 mg) was used.

Measurement was performed on three samples for each experimental treatment.

The durability index was determined in accordance with European standard UNE-EN ISO17831-2. Under this standard, a rotating drum of 598 mm in diameter and length is used, with an internal flange that lifts the briquettes internally and drops them during rotation. The test lasts 5 min and is carried out at a rotation speed of 21 rpm (UNE- EN ISO 17831-2, 2016; UNE- EN ISO 18134-3, 2016). The samples are extracted from the drum and are sieved using a mesh 2/3 of the diameter or diagonal of the briquettes. An Ohaus precision scale (EX1103, Precision Explorer of 1,100 g \times 1 mg) was used to weigh of the briquettes.

Compression strength test is executed according to the procedure established in the ASTM E9 standard (ASTM E9, 2018). Tests were achieved on universal test machine Shimadzu AGX PLUS of 300 kN equipment. A load is applied with a constant displacement setting of 5 mm/min. Three samples are tested for each experimental treatment. Figure 3 shows the assembly for execute the compression strength test.

2.5. Other characterization tests

Observations are made to bonding mechanisms among fibers to determine the arrangement by which they are achieved. For this, stereomicroscopy (magnification of 2x to 12.6x are set) and Scanning Electron Microscopy (magnification of 200x to 5000x are set) are used for the detailed microscopic analysis of the constituents of briquettes that show the better mechanical properties.

An elemental analysis is also carried out by means of an elemental organic analyzer using a Thermo Scientific Flash 2000 CHNS/O device, with the purpose of determine the chemical composition in terms of carbon, nitrogen, hydrogen, sulfur and oxygen, the latter by difference. An amount of 5 mg of V_2O_5 are added in the test to enhance the reaction and to allow a better detection of sulfur.

A test is performed for the determination of heating value, in accordance with the procedure described in ASTM D240 standard (ASTM



Figure 3. Experimental setup for compression testing of EFB briquettes. Source: Authors.

D240, 2017), which establishes a method for the measurement of combustion heat by means of a pump calorimeter. For this, a Mettler Toledo model AB 204 SNR 111660708 scale and IKA C 2000 Basic S1 calorimeter are used, operating in isoperbolic mode. Extra dry industrial oxygen grade 2.7 is used at a pressure of 30 bar. The temperature of the calorimeter jacket was controlled at 25 °C. In addition, analytical correlations are used to theoretically calculate the heating value of the samples to compare them with the experimentally obtained values.

Ash content measurements are made for the purpose of calculating heating value in accordance with the procedure described in ASTM E1755 (ASTM E1755, 2015). Moreover, the determination of the ash content is essential, given the importance attached to the standard of briquetting for energy purposes, by establishing allowed limit levels.

3. Results and discussion

3.1. Results for briquettes density

Results obtained from the measurements were tabulated and entered in statistical program to establish the effect of independent essential variables on the classification factors. A code written in the GNU Octave V5.1.0 software was used for this purpose. The response surface for briquettes density of EFB is shown in Figure 4. While, in Figure 5 the main effects for briquettes density considering the independent variables compaction time, moisture content and fiber length are show. From now on, W is the coded moisture content, FL is the coded fiber length and CT is the coded compaction time. For briquettes density the analysis has an adjusted correlation coefficient of 89.2% and an average absolute error of 37.5 kg/m³. From the graph inspection of the residues for briquettes density, it is estimated that they are distributed in the form of a centered band without showing asymmetry or a determined anomalous pattern.

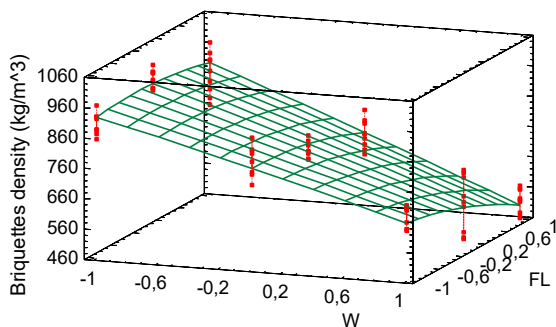


Figure 4. Response surface for briquettes density (compaction time set at 40s). Source: Authors.

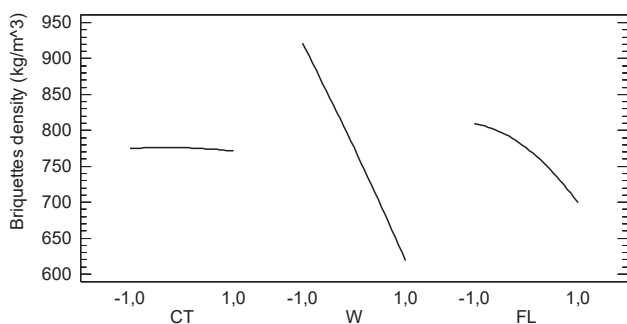


Figure 5. Main effects plot for briquettes density considering the experimental factors of compaction time, moisture content and fiber length. Source: Authors.

Table 2. ANOVA table for briquettes density. Source: Authors.

Source	Sum of squares	Mean square	F-Ratio	P-Value
CT	160.167	160.167	0.06	0.8042
W	1.24154E6	1.24154E6	480.14	0.0000
FL	162142	162142	62.70	0.0000
CT ²	140.747	140.747	0.05	0.8162
CT*W	8993.36	8993.36	3.48	0.0664
CT*FL	4853.44	4853.44	1.88	0.1751
W ²	593.21	593.21	0.23	0.6335
W*FL	33124.0	33124.0	12.81	0.0006
FL ²	8377.93	8377.93	3.24	0.0762
Total Error	178422	2585.82		
Total corr.	1.65702E6			

Additionally, from the lack-of-fit test it is concluded that the model fits experimental data.

Figure 5 shows that the variable with the greatest influence is moisture content, its linear behavior being descending. The highest values for briquettes density are obtained with moisture contents of 8% w.b. On the other hand, the second variable with the greatest influence is fiber length, its behavior being quadratic downward. The highest density value obtained was 934.4 kg/m³ for a fiber length of 74.7 mm, moisture content of 8% w.b. and compaction time of 40 s. Regarding compaction time, no statistically significant influence is observed.

Table 2 shows the results of the ANOVA test for briquettes density. Where, the F-ratio is the ratio between the mean square between groups and the mean square within groups and p-value is the probability of getting a result at least as extreme as the results observed during the test. A marked influence of moisture content can be observed, followed by fiber length and non-significant influence of compaction time for briquettes density response variable. There is also a significant influence of interaction among moisture content and fiber length, but there is no a significant influence of other interactions or quadratic dependence. Moisture content has 2.8 times greater influence on the results for briquettes density than fiber length factor. This is because moisture content determines the intensity of bonding mechanisms among biomass fibers, influencing the activation of lignin and the plasticization of densified biomass.

The adjusted regression equation for briquettes density model is also obtained from the statistical processing of the experimental data. After eliminating statistically non-significant coefficients, according to ANOVA, the expression of the empirical model for the behavior of briquettes density (D_b) based on the moisture content and the fiber length is as follows:

$$D_b = 775.3 - 151.6W - 54.8FL - 30.3W FL \tag{1}$$

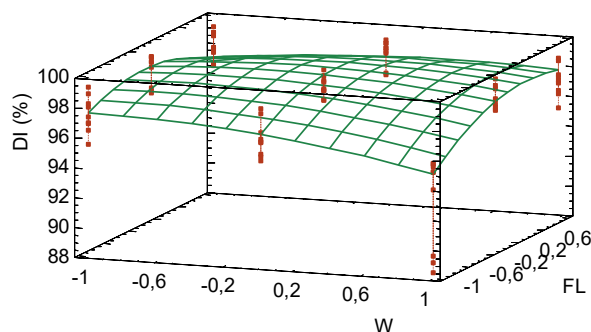


Figure 6. Response surface for the durability index (compaction time set at 40s). Source: Authors.

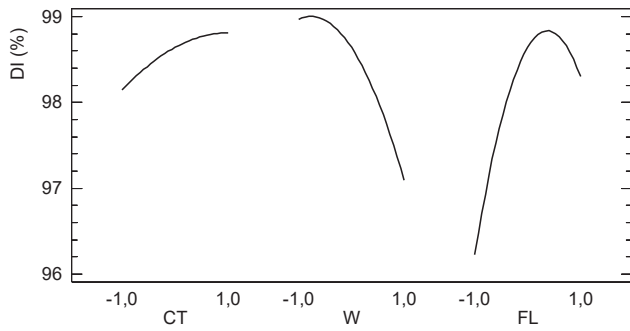


Figure 7. Main effects plot for durability index considering the experimental factors of compaction time, moisture content and fiber length. Source: Authors.

3.2. Results for durability index

In Figure 6 the response surface for durability index obtained according to experimental factors of moisture content and fiber length is appreciated. Figure 7 shows the main effects of the mechanical durability index for the independent variables studied, finding that the moisture content has the greatest effect on the mechanical durability index. For the durability index the analysis has an adjusted correlation coefficient of 65.4% and an average absolute error of 0.95%. From the graph inspection of the residues for the durability index, it is estimated that they are distributed in the form of a centered band without showing asymmetry or a certain anomalous pattern. Additionally, from the lack-of-fit test it is concluded that the model fits experimental data.

In Figure 7 it is observed that there is an influence of the experimental factors on the durability index a little different than found for briquettes density. The highest durability index value obtained was 99.05% for a fiber length of 92.2 mm, moisture content of 8.42% w.b. and compaction time of 6.8 s. There is not a statistically significant influence of compaction time observed for the durability index.

Table 3 shows the ANOVA analysis for the durability index. Similar effects of fiber length and moisture content on the durability index are statistically demonstrated. Additionally, the significant influence of several interactions is appreciated (CT*W; CT*FL and W*FL), followed of fiber length square. A possible explanation of the observed behavior is that the greater the fiber length, the entanglement of these is increased, thus avoiding greater detachment. However, if fiber length is greater than 100 mm, the durability index begins to decrease because the cross-linking between fibers is difficult for the nominal briquette diameter studied. There is also a more pronounced elastic restitution phenomenon of the briquette material. The latter was evidenced by the observation of briquettes several hours later the densification process.

The adjusted regression equation for the durability index as a function of coded moisture content, coded fiber length and coded compaction time is as follows:

Table 3. ANOVA table for the durability index. Source: Authors.

Source	Sum of squares	Mean square	F-Ratio	P-Value
CT	6.02002	6.02002	3.57	0.0630
W	47.4703	47.4703	28.15	0.0000
FL	57.9083	57.9083	34.34	0.0000
CT ²	0.464006	0.464006	0.28	0.6016
CT*W	9.0601	9.0601	5.37	0.0234
CT*FL	7.943	7.943	4.71	0.0334
W ²	6.66125	6.66125	3.95	0.0508
W*FL	27.1094	27.1094	16.08	0.0002
FL ²	34.1689	34.1689	20.26	0.0000
Total Error	116.362	1.68641		
Total corr.	336.819			

$$DI = 98.64 - 0.938W + 1.036FL + 0.502CT W - 0.469CT FL + 0.868W FL - 1.378 \cdot FL^2 \tag{2}$$

3.3. Results for compressive strength

Figure 8 shows the curves of compression versus deformation for the briquette group that uses a fiber length of 85 mm. It is possible to appreciate that the average maximum compressive strength is 2,500 kgf with a deformation of briquettes of 30 mm.

The response surface for compressive strength is shown in Figure 9, where it is seen that moisture content remains the most influential experimental factor followed by fiber length. Figure 10 shows the main effects of the experimental factors on the maximum compression strength. For compressive strength the analysis has an adjusted correlation coefficient of 82.1% and an average absolute error of 4.26 kN. From the graph inspection of residuals for compressive strength, it is estimated that they are distributed in the form of a centered band without showing asymmetry or a certain anomalous pattern. Additionally, from the lack-of-fit test it is concluded that the model fits experimental data.

Table 4 shows the results for ANOVA analysis for compressive strength. Where it is appreciated that all experimental factors have a significant influence on the behavior of compressive strength. Additionally, a significant influence of the interactions of compaction time and fiber length (CT*FL) and compaction time and moisture content (CT*W) are observed. It is concluded that ideal values to achieve the highest compressive strength are moisture content of 8% w.b., fiber length 50 mm and compaction time of 15.3 s. For these reference values of experimental factors, a compressive strength of 44.54 kN is achieved.

The adjusted regression equation for the compression strength model is also obtained from the statistical processing of experimental data. After eliminating the statistically non-significant coefficients, according to ANOVA, the regression model for compression strength based on moisture content, fiber length and compaction time is as follows:

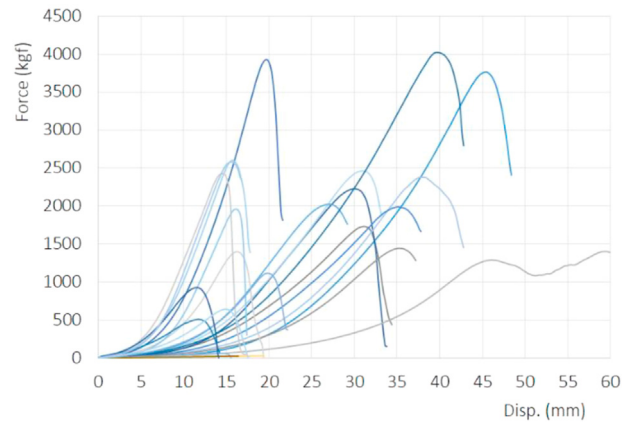


Figure 8. Behavior of compression force vs. stroke for briquettes made of 85 mm length fibers. Source: Authors.

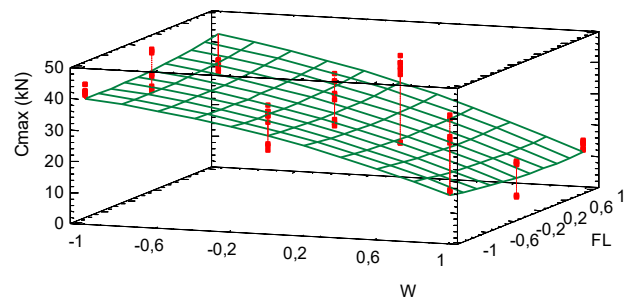


Figure 9. Response surface for compressive strength (compaction time set at 40s). Source: Authors.

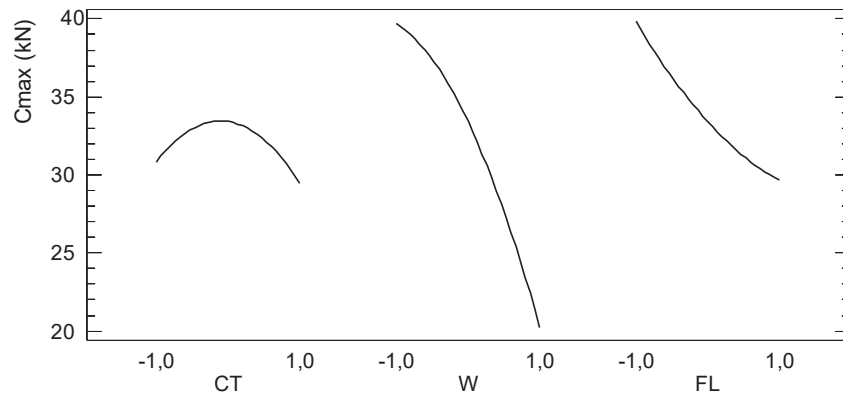


Figure 10. Main effects for compressive strength considering the experimental factors of compaction time, moisture content and fiber length. Source: Authors.

Table 4. ANOVA table for compressive strength. Source: Authors.

Source	Sum of squares	Mean square	F-Ratio	P-Value
CT	26.8535	26.8535	0.97	0.3273
W	5124.8	5124.8	185.75	0.0000
FL	1399.55	1399.55	50.73	0.0000
CT ²	194.001	194.001	7.03	0.0099
CT*W	653.569	653.569	23.69	0.0000
CT*FL	934.423	934.423	33.87	0.0000
W ²	214.199	214.199	7.76	0.0069
W*FL	109.237	109.237	3.96	0.0506
FL ²	31.725	31.725	1.15	0.2873
Total Error	1903.68	27.5896		
Total corr.	10613.2			

$$C_{max} = 33.39 - 9.742W - 5.091FL - 3.283CT^2 - 3.449W^2 - 4.261CTW + 5.095CTFL \tag{3}$$

3.4. Multiple response optimization

Each studied answer reaches its optimum for a combination of the values of the experimental factors that are different. Therefore, it is appropriate to define the best operating point using a composite desirability function. The goal is to maximize the overall desirability function and equal weighting is used for each of the three experimental responses. In Figure 11 the response surface of the composite desirability function is shown.

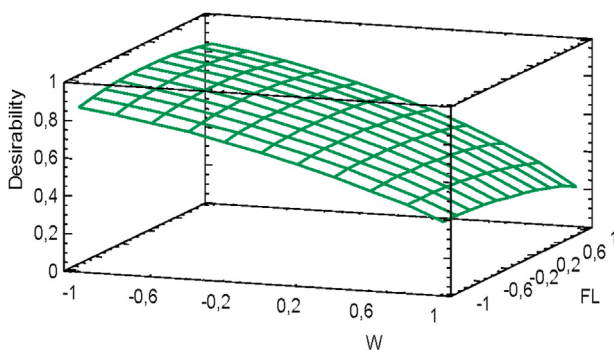


Figure 11. Response surface of the composite desirability function. Source Authors.

In Table 5 the best point of operation is shown, where the maximum for the desirability function is obtained, $D = 0.912$. These reference values for the experimental factors should be used to achieve an optimal and balanced composite behavior between the three dependent experimental variables that characterize the quality of the briquettes in this study. A validation of the experimental reference values for the best operating point was carried out; for this, five experimental runs were carried out. It was found that the average values for briquette density was 910 kg/m^3 , durability index 98.90% and finally maximum compression 40.5 kN. The foregoing confirms that reference values for experimental factors achieve a balanced compromise of the best integral response.

3.5. Results of observations using stereomicroscopy

Figure 12 shows stereo microscopy images obtained for briquettes with the best mechanical properties, according to the results of statistical processing of experimental data. From the detailed observation of these images it can be concluded that the length of the fibers influences the mechanical bond. For short fiber length there is a greater accommodation and little fiber spacing, while for long lengths a greater cross-linking of fibers is observed. Regarding the mechanism of action of moisture, there is no clear influence on the increases used, so observations with other techniques are required.

3.6. Results of observations using scanning electron microscopy

Scanning Electron Microscopy is performed with magnifications between x200 and x5000 to establish shape and cause of union among fibers due to differences in moisture content. Figure 13 shows that for a moisture content of 8% w.b. there is a greater "welded area" or fusion

Table 5. Experimental factor values to maximize the global desirability function. Source Authors.

Factor	Coded value	Real value
CT	0.328	26.6 s
W	-1.0	8%
FL	-0.324	73.6 mm

between fibers compared to that observed for a moisture content of 16% w.b. At higher magnifications (x1000 and x5000) small welding points can be seen joining the empty spaces between fibers. When these SEM images are compared with those reported by other authors (Yan et al., 2015; Gong et al., 2016), it can be presumed that the whitish clusters are activated lignin. However, complementary magnetic resonance studies are required to confirm this issue.

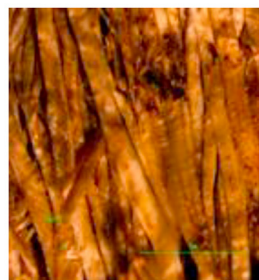
It is concluded that there is a first mechanism of purely mechanical union, through cross-linking and blocking among fibers. This is aided by "weld points" created in adjacent fibers where the activated lignin acts as a binder, in addition to other secondary bonding mechanisms such as H bridges (Anuar et al., 2019). It is observed that a high moisture content generates some relative distance among fibers, less activation of lignin and consequently less cohesion among fibers, which coincides with what was found in (Arzola et al., 2012, 2014). Likewise, for a longer time of compaction, areas with wider lignin activation and a better packing of fibers inside a briquette are obtained (Medupin et al., 2017).



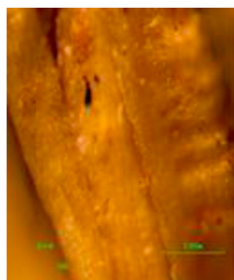
8% w.b., 85 mm and 20 s (2x)



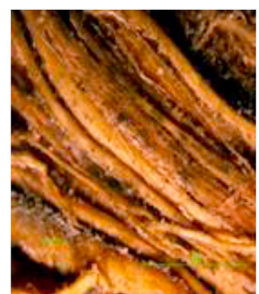
8% w.b., 85 mm and 20 s (12.6x)



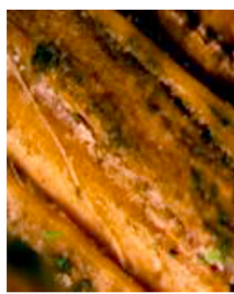
8% w.b., 50 mm and 40 s (2x)



8% w.b., 50 mm and 40 s (12.6x)



16% w.b., 85 mm and 20 s (2x)



16% w.b., 85 mm and 20 s (6x)

Figure 12. Stereo microscopy images for some samples. Source Authors.

3.7. Results for chemical composition and higher heating value

Table 6 shows the chemical composition for the three crops using elemental analysis. This chemical composition is used for the theoretical calculation of the heating value of biomass, and subsequent contrast with the experimental results of heating value obtained directly. The purpose of these tests is to determine the chemical composition and the higher heating value for the EFB African oil palm and then find the degree of suitability of this biomass to be used as briquettes for energy purposes. It also seeks to determine if there are differences in terms of the aforementioned characteristics according to the region of origin of the biomass.

Table 7 shows the results of measurements of higher heating value (HHV) for each crop. Standing out, the fact that there are no substantial differences regarding heating value depending on the region of origin, adding that heating value is between 17.84 MJ/kg to 18.63 MJ/kg.

Table 8 shows the results obtained from theoretical calculation of heating value using correlations by proximate and ultimate analyses, using the data obtained from the chemical composition by elementary analysis (Medupin et al., 2017; Sheng and Azevedo, 2005).

The ash content (%Ash) measured has an average result of 5.62% for biomass from the three regions. About the generation of ashes, it can be affirmed that briquettes made with EFB African oil palm are within the acceptability specifications of European Standards. Table 9 shows the correlation of Sheng and Azevedo (2005), used as an additional means to estimate the higher heating value through the ash content.

Table 8 shows that there is a similarity with the results obtained experimentally. The crop with the highest heating value is C2 (Sabana de Torres), independent of the correlation and that the lowest heating value occurs for crop C3 (Puerto Gaitán). This is due to carbon differences found in the elementary analysis for crops.

On the other hand, less than 1% of sulfur was detected in the samples tested. From which it can be concluded that very few SO_x emissions would be generated during combustion for energy production using EFB briquettes.

4. Discussion

In this study, bulk density obtained for EFB was 934.4 kg/m³, meanwhile durability index was 99.05% for the following levels of experimental factors, moisture content of 8% w.b., particle size of 85 mm, compaction time of 40 s, compaction pressure of 62 MPa and pre-heating temperature of 90 °C. On their behalf, Kaliyan y Morey, informed that bulk density of biomass coming from maize straw similar to EFB is 1100 kg/m³, and durability of 90%, with moisture content of 10% w.b., pressure of 150 MPa, particle size of 3 mm and preheating temperature of 85 °C (Kaliyan and Morey, 2010). Table 1 provides moisture content, fiber length and compaction time used values. In the biomass from corn, percentages of lignin, cellulose and hemicellulose are 40%, 41% y 5.8% respectively (Kaliyan and Morey, 2010; Wattanachira et al., 2016), while percentages in the EFB of cellulose are 34%, of lignin 21% and hemicellulose of 17% approximately (Nakagawa-Izumi et al., 2017; Galwango et al., 2019). Cellulose, lignin and hemicellulose are known as "natural binders" in the densified biomass; these can be activated both by softening (moisture content) as well as the effect of temperature (pre-heating) (Kaliyan and Morey, 2010; Zvicevičius et al., 2018). Kaliyan and Morey, conclude that lignin and hemicellulose are thermoplastic materials that present plastic deformations at low pressures and softening temperatures, this deformation being the cause of the permanent union between the particles, by producing then cohesion that increases density. Furthermore, the moisture content present in the biomass acts as a bridge and allows the cohesion of particles for distances between 0.5-0.74 nm, which corresponds to twice the length of the hydrogen bond (Kaliyan and Morey, 2010; Obernberger and Thek, 2004). Figure 13 shows the bond among particles. The differences among the results of the two biomasses for density and durability are based on the difference in structure present

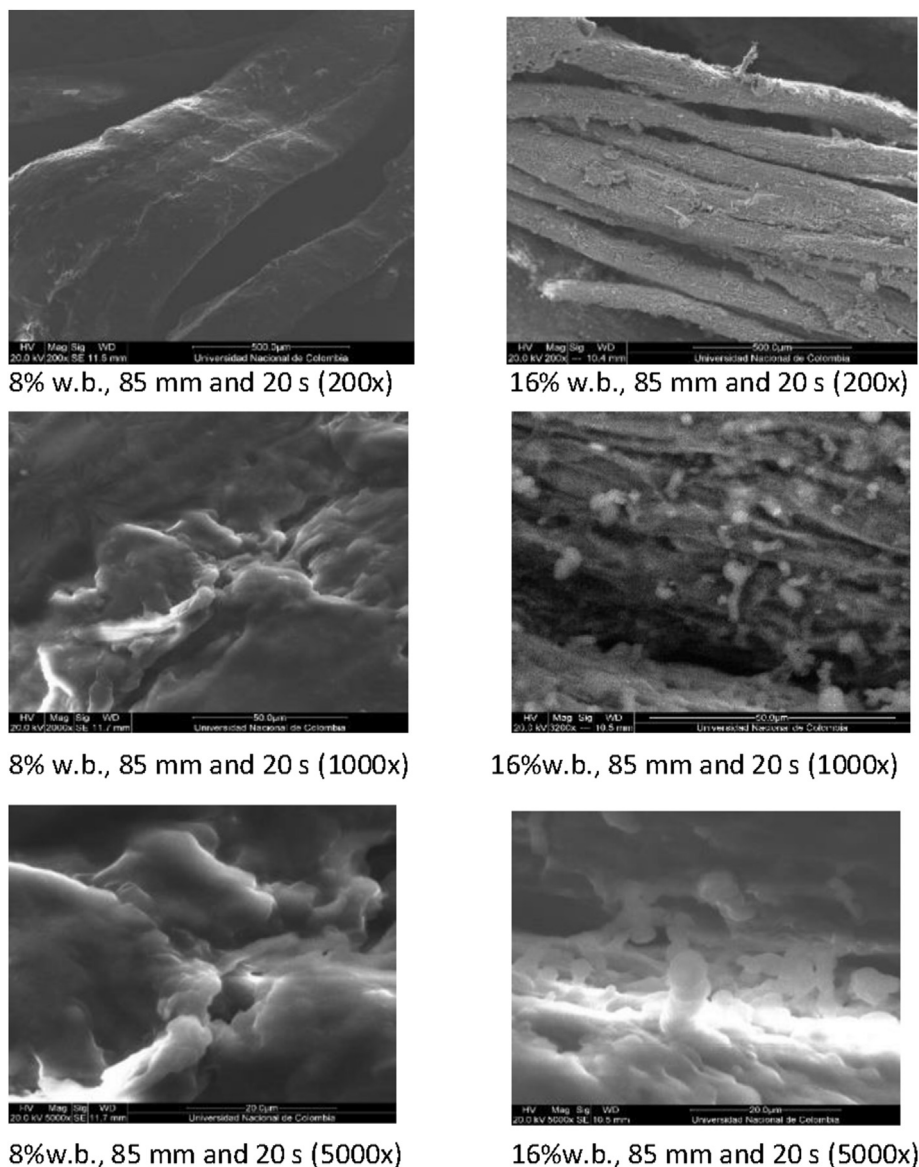


Figure 13. Scanning electron microscopy images for some samples. Source: Authors.

Table 6. Chemical composition of the samples (% mass) by region of origin. Source: Authors.

Chemical element	Region of origin		
	C1	C2	C3
Carbon	40.81	47.69	37.76
Hydrogen	5.67	6.20	5.97
Nitrogen	1.01	1.04	1.01
Total	47.49	54.93	44.75

Table 7. Heating value per crop. Source: Authors.

Region of origin	Higher Heating Value [MJ/kg]
C1	18.63
C2	17.84
C3	18.19

in the fibers. While in the EFB its structure is cylindrical with small diameters - bar-type structures -, in the biomass from corn, its structure is of the laminar type. The latter, for being laminar and having a smaller particle size, by a factor of 0.3, decreases the spacing to form the pellet in a more effective way, being its density higher. However, the durability is less because the fibers with a greater length of the EFB, regarding corn biomass, allows an interweaving among them, while the laminar structure does not allow such a high a density of interlinking and mechanical “closures,” decreasing its durability index with respect to EFB by approximately 9%.

Table 8. Prediction of heating value (HHV) [MJ/kg] using ultimate analysis. Source: Authors.

Empirical correlations	Region of origin		
	C1	C2	C3
Tillman	16.176	19.183	14.844
Sheng and Azevedo	16.759	19.001	15.767
Boie	15.159	18.196	14.442
Graboski and Bain	16.651	20.005	15.358
Jenkins	17.856	19.731	17.273

Regression models obtained in this research partially explain the behavior of the responses to briquette density, durability index and strength to compression according to defined experimental factors. The values of the adjusted correlation coefficients reflect that there is a part of the variation of the response variables that cannot be explained by means of the three experimental factors used in this research. Furthermore, the standard deviation of residuals (σ_R) for the three regression models, although small, are of appreciable relative magnitude. This is evident if the relative size (in percentage) of the standard deviation of residuals is determined with respect to the difference between the maximum value and the minimum expected value from the responses obtained by means of regression models, or with respect to the independent coefficient value of regression models. In Table 10 the above can be observed, in which the percentage of variation with respect to the ranges (maximum predicted value minus minimum predicted value of each response) fluctuates between 11.77% and 19.23%; while, in relation to independent coefficients of regression models, the relative variation of standard deviation of residuals turned out to be between 1.31% and 15.72%.

In the ANOVA analysis, shown in Tables 2, 3 and 4, it is shown that density, durability index and compressive strength respectively, are significantly affected by the moisture content and the length of the fiber. Furthermore, there is an influence of the interaction between compaction time with fiber length and moisture content, similarly between moisture content and fiber length ($p < 0.05$). The results of the effects on density and durability of the variables of moisture content and particle size agree with those obtained in the ANOVA analysis performed by Kaliyan and Morey; with the difference that Kaliyan and Morey use a general univariate model to understand the main effects (Kaliyan and Morey, 2010), whereas in this research response surfaces were used in order to visualize nonlinear (quadratic) behaviors in the main effects.

Zvicevičius et al. highlight that, for biomass from wood, as humidity decreases to values close to 10% w.b., density is increased, and then for a humidity below 10% w.b. there is no significant influence on density (Zvicevičius et al., 2018). The previous behavior was demonstrated in the preliminary tests carried out at EFB with a moisture content of 4% w.b., in which the non-relevance of the response variables is found when using

Table 9. Indirect calculation of higher heating value (HHV) [MJ/kg] by proximate analysis. Source: Authors.

Correlation of Sheng and Azevedo	Heating value	Correlation coefficient
$19.914 - 0.2324 \cdot \%Ash$	18.607	0.625

Table 10. Relative behavior of the standard deviation of residuals (σ_R). Source: Authors.

Response variable	Maximum pred. value	Minimum pred. value	Model constant (C)
Briquette density (kg/m ³)	934.4	502.5	775.3
Durability index (%)	99.05	92.34	98.64
Compression strength (kN)	44.54	11.28	33.39
Response variable	σ_R	$\sigma_R / (\text{MaxPV} - \text{MinPV}) \times 100\%$	$\sigma_R / C \times 100\%$
Briquette density (kg/m ³)	50.85	11.77	6.56
Durability index (%)	1.29	19.23	1.31
Compression strength (kN)	5.25	15.78	15.72

the lower level of the moisture content. On the other hand, Zvicevičius et al. report a little relevance of the compaction pressure in the density results, the above corresponding to the behavior found in preliminary tests varying the pressure for EFB. On the other side, from the preliminary tests it is established that increases in moisture content lead to a decrease in the compression ratio, which is consistent with what was found by others researchers; where the optimal moisture content for the corn biomass was 9.4% w.b. (Zvicevičius et al., 2018) close to that found for the EFB, which was 8.4% w.b. Furthermore, according to Jackson et al., moisture content has an inversely proportional effect on the variation of durability index. This agrees with the behavior shown in Figures 6 and 7, in which it is observed that increases in moisture content produce a downturn in the durability index (Jackson et al., 2016).

In another work the researchers (Frodeson et al., 2019) established that the influence of the iteration between moisture content and compaction time is based on the reduction of the compression pressure. A greater presence of "natural binders", which can be activated at low densification pressures, allows the increase of permanent cohesion using a softening temperature around 90 °C for biomass from wood. This behavior is also exhibited in EFB, in which the iteration between moisture content and compression time is not relevant as shown in the results of the ANOVA analysis presented in Table 2. The relevance of this iteration is only presented in the decline of compaction pressure.

On the other hand, the highest calorific value found in the EFB was 18.6 MJ/kg with a compaction pressure of 62 MPa and a temperature of 90 °C, as shown in Tables 7 and 9. In preliminarily manufactured test tubes varying pressure compaction and temperature, it was found that the increase in densification pressure has a proportional effect on the calorific value, while temperature does not show a significant influence, which agrees with other reported results (Tang et al., 2016).

Additionally, in the present study Scanning Electron Microscopy (SEM) is performed on the EFB fibers after the densification process, which can be compared with SEM images obtained by other researchers. The analysis of images obtained by optical microscopy and scanning electron microscopy allowed to understand a little better the mechanisms of fusion among biomass fibers. In the first place, a mechanism of mechanical union is evidenced, by cross-linking and blocking among fibers. Which is aided by "weld points" created in adjacent fibers where activated lignin acts as a binder and other secondary cohesion mechanisms such as H bridges. It is observed that a high moisture content generates a certain relative distance among fibers and a smaller lignin activation, consequently, less cohesion among fibers. Likewise, for a longer time of

compaction, areas with wider lignin activation and a better packing of fibers inside the briquette are obtained. Figure 13 shows cluster or joined structures without a defined measurable length or diameter. Moreover, α -cellulose with interlaced nonuniform distributions is evidenced, which agrees with other reported results (Galiwango et al., 2019). In this latest research, structures made up of rod-shaped microfibrils are identified, in which the diameters of these fibers can be differentiated.

5. Conclusions

In this work, statistical empirical models are obtained, which partially explain the influence of moisture content, fiber length, and compaction time in different parameters that characterize quality and acceptability of EFB of African oil palm briquettes, based on a set of mechanical and thermal responses. Regarding the influence on mechanical properties of experimental factors, there is a great influence of moisture content on briquettes density. In addition, to obtain a high density it is desirable to use short fibers. On the other hand, with respect to durability index, the experimental factor that has the greatest influence is fiber length closely followed by moisture content. Compressive strength of briquettes was affected by moisture content, fiber length, compaction time and interactions between compaction time and fiber length, and compaction time and moisture content. From this experimental investigation it can be concluded that the best briquette of EFB of African oil palm is obtained with a moisture content of 8% w.b., 73.6 mm fiber length and a compaction time of 26.6 s. With previous levels of experimental factors, together with pressure and temperature established for the densification process, a correct balance is obtained among mechanical properties, reaching a good mechanical performance and an adequate target of heating value of the densified biomass. Finally, it can be concluded that briquettes made of EFB African oil palm, with the best mentioned values of experimental factors, comply with the briquettes international standards used to date.

Declarations

Author contribution statement

Huber Cabrales: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data.

Nelson Arzola: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

Oscar Araque: Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

Funding statement

This research did not receive any specific grant from funding agencies in the public, commercial, or not-for-profit sectors.

Data availability statement

Data included in article/supplementary material/referenced in article.

Declaration of interests statement

The authors declare no conflict of interest.

Additional information

No additional information is available for this paper.

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