PAPER 90 – OPTIMIZATION OF CHARCOAL BRIQUETTES PRODUCTION PROCESS FROM RAFFIA PALM (Raphia Hookeri) EPICARP USING RESPONSE SURFACE METHODOLOGY

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ABSTRACT

In this study, charcoal briquettes were produced from carbonized raffia palm fruit shells using synthetic gum and cassava starch as binders. Response Surface Methodology (RSM) using rotatable Box-Benkhen Experimental Design (BBD) was used to optimize the briquette quality. The briquette samples were made by compressing the carbonized raffia palm fruit shells in an existing briquetting machine. The factors considered in the study were particle size, binder-substrate ratio, and drying time while relaxed density and the calorific value of the briquettes were the response variables. Statistical models were developed to predict the various responses. Results of the study indicated that a combination of 1.05 mm particle size, 29.41 % binder ratio, and 3 days drying time of synthetic gum bonded briquettes was optimum. Under the optimum settings of factors, the synthetic gum-bonded briquettes had a relaxed density of 451.99 Kg/m³ and a calorific value of 21535.2 kJ/kg. The results also revealed that a combination of 1.99 mm particle size, 10 % binder ratio, and 3 days drying time of starch-bonded briquettes were optimum. Under the optimum settings of the factors, the starch-bonded briquettes had a relaxed density of 474.02 Kg/m³ and a calorific value of 20185.5 KJ/kg. Validation of the models indicated that there was no significant difference between the predicted and experimental values which implied that the models could reliably predict the responses.

KEYWORDS: Briquettes, Box Benkhen Design, Response Surface Methodology, Modelling & Optimization, Raffia Palm Epicarp.

1. INTRODUCTION

Briquetting is a process of binding together pulverized carbonaceous matter, often with the aid of a binder (Martin et al., 2008). The common forms of briquettes are coal briquettes and biomass briquettes. Biomass briquettes originate from mostly agricultural residues. Briquettes may be produced with or without binders. The particles must bind properly during compression; otherwise, the briquettes might crumble easily. Pressure, heat, moisture, and size reduction are therefore necessary in the process of making high-quality briquettes. The binders can be organic or inorganic agents. Some of the identified binders of organic nature are heavy crude oil, starch, and molasses. The inorganic binders include clay, sodium silicate, and cement. The binder types, amount of binder agent and water addition, have significant effects on the thermal behaviour and combustion of the briquettes (Altun et al., 2003). The combustion characteristics of the briquettes may be determined based on different parameters such as the reaction kinetics and the water boiling tests.

Most of the technologies for briquetting are expensive and unaffordable by the rural people, who need them most. It is pertinent, therefore, to design briquetting machines which are efficient but less expensive for the end users. In the current work, Raffia palm shells, an abundant agro-biomass in the tropics were carbonized using a laboratory muffle furnace for ease of experimentation. However, for the implementation of the technique in a rural setting, the authors proposed and tested the suitability of employing the drum method for the carbonization of the sample biomass material, while using the same biomass (raffia palm shells) as the primary energy source for carbonization. The briquette production process was done using a developed machine.

Although studies on the production of briquettes from agricultural waste biomass and its optimization using response surface methodology have been reported for rice husk (Musabbikhah *et al.*, 2017), palm kernel shells (Ugwu and Agbo, 2011), the mixture of Acarcia bark and Palm Kernel Shells (Kurnianwan *et al.*, 2017), corn cob residue mixed with macadamia shells (Suminya and Suchana, 2017), while studies on the production charcoal briquettes from Raffia Palm shells have only been reported in the literature by Iwar *et al.* (2018). To the best of the authors' knowledge, no previous studies have been conducted on the production and optimization of charcoal

briquettes from raffia palm shells using response surface methodology, which justifies the novelty of the current study.

Mathematical optimization is defined as the selection of best factors or elements (with regard to some set goals or constraints before the beginning of the selection) among some group of factors and elements considered. More generally, optimization includes finding the "best available" values of some objective function given a defined domain (or input), including a variety of different types of objective functions and different types of domains (Battiti *et al.*, 2008). Several mathematical approaches have been used to achieve optimization including Response Surface Methodology (RSM).

RSM can be described as a technique that involves complex calculations for the optimization process. This approach develops a suitable experimental design that integrates all of the independent variables and uses the data input from the experiment to finally come up with a set of equations that can give a theoretical value of an output. The outputs are obtained from a well-designed regression analysis that is based on the controlled values of independent variables. Thereafter, the dependent variable can be predicted based on the new values of independent variables. RSM involves the use of the following experimental designs: Box-Benkhen Design (BBD); Central Composite Design (CCD); Optimal Designs etc. (Khairul and Mohammed, 2015). For this study, the Box Benkhen design is preferred due to its flexibility and reduced experimental runs and its rotatable ability when blocking is not required.

The significance of this study lies in the fact that the optimal and most valid model can be used to predict the increase or decrease in the values of every dependent variable. Therefore, this work will contribute to giving significant impacts on society, by aiding the production of high-quality renewable fuel. Furthermore, the findings of this research could be seen as a step in the right direction toward curbing the menace of deforestation for the purpose of charcoal production and energy supplies.

2. MATERIALS AND METHODS

This section was divided into two namely: the briquetting process and experimental design.

2.1 Briquette Production Materials and Process Description

The materials and equipment used for the study include the fabricated briquette machine, black coated and covered drum (carbonizer), raffia palm shells, cassava starch, synthetic gum, bomb calorimeter, muffle furnace, set of mechanical sieves, electronic weighing balance, measuring cylinders, pressure gauge, vernier callipers, laboratory mortar and pestle and a mechanical hammer mill. Numerical designs and detailed descriptions of the machine are reported elsewhere in our previous study (Iwar *et al.*, 2018).

2.2 Biomass Feed Stock Preparation and Carbonization

Raffia palm fruits were collected from the neighbouring bushes in Nasarawa State Nigeria. The fruits collected were crushed and dried in the sun to reduce the moisture content of the feedstock to ensure effective carbonization. The dried raffia palm shells were shredded to small sizes using a laboratory mortar and pestle to provide more surface area for carbonization. A muffle furnace model SXL produced by Gallenkamp England, was used for the carbonization of the dried raffia palm shells. The biomass (raffia palm shells) was fed into the muffle furnace at a manageable batch of 100g at a time. 100g was weighed using a digital electronic weighing scale and put into crucibles. The crucibles were then loaded into a muffle furnace and ignited at a pre-set temperature of 450°C for 45 min. The crucible and contents were unloaded and placed in desiccators to fully cool down as recommended by Martin *et al.*, (2008). The carbonized Raffia palm shells were then put in well-labelled plastic bags to avoid absorbing moisture from the atmosphere and were then ground into fine particles using laboratory mortar and pestle and kept for further processing.

2.3 Binder Preparation and Briquette Production Procedure

For the purpose of this research work, two binders were used, cassava starch (natural binder) and synthetic gum (synthetic binder). At first, the cassava starch of 0.25kg was dissolved in 0.5 litre of cold water to form a paste, and 2.5 litre of water was put to boil. The paste was gradually mixed with the boiling water and stirred gently

while hot until a smooth homogeneous gelatinized starch solution was obtained. The temperature of the stove heating the mixture was kept constant throughout the process and the mixture was continuously stirred for 5 to 10 minutes, until it turned slightly lumpy and rather viscous.

The pulverized carbonized raffia palm shells were then sieved using a mechanical sieve shaker into three different particle sizes of 0.3 mm, 1.15 mm, and 2.0 mm sieves. The different particle sizes were then put in labelled plastic bags for proper identification. The cooked cassava starch was poured into a mixer containing pulverized carbonized raffia palm shells at 10 %, 30 %, and 50 % binder levels and agitated properly to enhance proper blending prior to compaction because the optimum binder level required to produce briquettes with desirable properties was to be determined. Synthetic gum produced by dissolving a known weight (g) of waste polystyrene in a known volume (mL) of gasoline (synthetic binder) was prepared in the same way and mixed with the biomass sample as above. The biomass-binder mixture was manually fed into the moulds of the fabricated machine and was compressed using a 20-ton hydraulic jack (Iwar *et al.*, 2018). The compressed briquettes were then left to set for about five minutes according to the method of Osarenmwinda and Ihenyen, (2012), after which the lid of the machine was opened for ejection and sun-drying of the briquettes for 3 – 7 days to explore the effect of drying on the briquette physical and thermal properties (Fig. S1as provided in the appendix). The flow chart of the briquette production process is presented in Fig. S2 (See appendix).

2.4 Determination of Briquette Physical Characteristics

The relaxed density (density determined when dried) and relaxation ratio (ratio of compressed density to relaxed density) of the briquette was determined in the dry condition of the briquette after sun drying to a constant weight at an ambient temperature. The relaxed density was then calculated as the ratio of the briquette weight (kg) to the new volume (m³). This indicates the relative stability of the briquette after compression (Oladeji, 2010). Similarly, the calorific values of the produced briquette samples were determined using Mahler-cook Bomb Calorimeter according to ASTM E711-87 standards (ASTM, 2004).

2.5 Experimental Design and Data Analysis

Box-Benkhen Design with 17 runs consisting of 12 actual design points and 5 centre points without replication for a rotatable design in Response Surface Methodology (RSM) was used to model the experimental factors and correlate the results with the responses (Marcos $et\ al.$, 2008). The factors considered were particle size, binder ratio, and drying time at three levels each. The responses were; relaxed density and calorific value of the briquettes. ANOVA tests at p=0.05 level of significance were performed on the experimental results and significant factors and interactions were identified. The optimum factor conditions were also determined and verified experimentally. Quadratic models were developed to predict and correlate the responses. The same approach was used for briquette produced using both synthetic and organic binders.

3. RESULTS AND DISCUSSION

3.1 Modelling Experimental Factors of Briquette Production

Box-Benkhen Design with 17 runs in Response Surface Methodology (RSM) was used to model the experimental factors and correlate the results with the responses as shown in Tables S1 and S2 in the supplementary material (Appendices) for the synthetic gum and starch-based briquettes respectively. The factors considered were particle size at three levels (0.30 mm, 1.15 mm, and 2.0 mm), binder-to-substrate ratio at 10 %, 30 %, and 50 %, and drying time at 3 days, 5 days, and 7 days. The responses were relaxed density and calorific value of the briquettes produced.

3.1.1 Relaxed Density of the Produced Briquettes

From the analysis of Variance (ANOVA) carried out on the briquette samples, it was observed that particle size, binder ratio, and drying time had significant effect on the relaxed density of the briquette produced ($P \le 0.05$). The ANOVA results also revealed that the interaction between the particle size and binder ratio on the relaxed density of the gum Arabic-bonded briquettes was significant, but the interaction between the particle size and drying time did not have significant effects on the relaxed density of the briquette as well as the interaction between the binder ratio and the drying time ($P \le 0.05$)

Box Benkhen Design was used to relate particle size, binder ratio, and drying time as well as their interaction to develop a second-order regression model (equation) that can be used to evaluate the relaxed density as a response as shown in equation (1).

(1)

Where represent relaxed density, particle size, binder ratio and drying time respectively.

The studentized actual versus predicted values plot (Fig. 1A) is almost a straight line showing that the model can predict the response. The plot shows that the predicted values coincide with the model values and the points that were outside the model line were negligible signifying the model's adequacy. Thus the model is significant and can be used the move around the regions of the experimental design.

Similarly, it was observed that the model, particle size, binder ratio and drying time of the briquette produced using starch as binder had significant effect on the relaxed density of the briquettes ($P \le 0.05$). A quadratic model for predicting the relaxed density of the briquettes produced with starch as binder was also developed and is as presented in equation (2). The model equation in terms of actual factors can be used to make predictions about the response (relaxed density) for given levels of each factor. The model equation is useful for identifying the relative impact of the factors by comparing the factor coefficients.

(2)

Where represent relaxed density, particle size, binder ratio and drying time respectively.

The studentized actual versus predicted values plot (Fig. 1B) is almost a straight line indicating that the model can predict the responses, thus errors in the model prediction ability may be due to noise. The plot shows that the predicted values coincide with the model values. The distance of the points from the model line is also lean, indicating that prediction error(s) in the model are negligible, which signifies the suitability of the model in predicting the response.

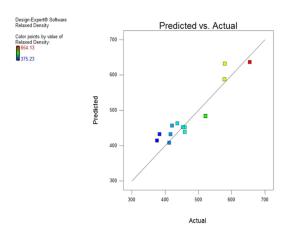


Figure 1 (A): Actual Versus Predicted Values plot for Relaxed Density of Synthetic Gum Based Briquettes

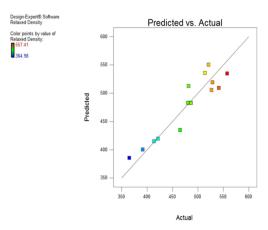


Figure 1 (B): Actual versus Predicted Plot for Relaxed Density of Starch Based Briquettes

3.1.2 Calorific Value of the Produced Briquettes

The results of ANOVA for the experiment on the calorific value of briquettes produced with synthetic gum revealed that the model and the binder ratio of the briquettes produced have significant effects on the calorific value of the briquettes, while the particle size and the drying time of the briquette had no significant effect on the calorific value of the briquettes ($P \le 0.05$).

The results were used to develop a linear model for predicting the calorific value of the briquettes as given in equation (3). The model equation in terms of actual factors can be used to make predictions about the response (calorific value) for given levels of each factor. The model equation is useful for identifying the relative impact of the factors by comparing the factor coefficients.

(3)

Where represent the calorific value, particle size, binder ratio and drying time respectively.

The "Externally Studentized Residuals" plot (Fig. 2A) is almost a straight line indicating that the model has the ability to predict the responses, the error in the model was insignificant. The plot shows that the predicted values coincide with the model values and the points that are outside the model line are not much as well as the distance of the points from the model line, therefore the errors in the model are negligible, which means the model is significant. The studentized residual plots vs run plots are used to check the model accuracy. From Fig. 2A, it could be seen that the residuals for each run (1-17) were within the acceptable limits from the predicted values, which indicates that the model developed could accurately predict the responses based on the studied factors (independent variables).

Similarly, the results of calorific values of briquettes produced with starch binder reveal that the model and binder ratio have significant effects on the calorific value of the briquettes, while the particle size and the drying time of the briquette have no significant effect on the calorific value of the briquettes at $p \le 0.05$ level of significance.

The results were also used to develop a linear model for predicting the calorific value of the briquettes as given in equation (4). The model equation in terms of actual factors can be used to make predictions about the response (calorific value) for given levels of each factor. The model equation is useful for identifying the relative impact of the factors by comparing the factor coefficients.

(4)

Where represent calorific value, particle size and binder ratio respectively.

Similarly, the "Studentized actual versus predicted" values plot (Fig. 2B) was almost a straight line showing that the model can predict the responses, with insignificant model errors. The plot shows that the predicted values coincide with the model values and the points that are outside the model line are not much as well as the distance of the points from the model line, therefore any errors in the model are negligible.

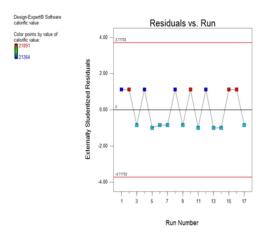


Figure 2 (A): Externally Studentized Residuals plot for calorific Value of Synthetic Gum-based Briquettes

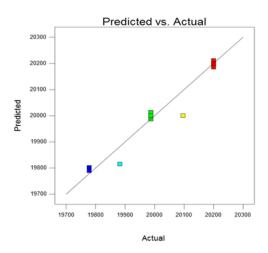


Figure 2 (B): Actual Versus Predicted Values plot for Calorific Value of Starch Based Briquettes

3.2 Optimization of the Briquette Quality

The result for the optimization reveals that a combination of the optimum values of particle size of 1.06mm, binder ratio of 29.41 % and the drying time of 3 days gives predicted values for the relaxed density and calorific value of the briquette produced with synthetic gum binder to be 451.99 kg/m³ and 21535.3 KJ/kg respectively (Fig. S3 in appendix). The value of desirability at the optimum conditions was found to be 0.524, which is within an acceptable range of 0.50 to 0.90 in accordance with Munoz-Hernandez *et al.*(2004).

Similarly, for the starch-based briquettes, the results for the optimization revealed that a combination of the optimum values of particle size of 1.99 mm, binder ratio of 10 % and the drying time of 3 days gives predicted values for the relaxed density and calorific value of the briquette produced with the starch binder to be 474.01 kg/m³ and 20185.5 KJ/kg respectively.

3.3 Experimental Validation

The correctness of the Box-Benkhen design (BBD) in design expert software was evaluated by producing briquettes from Raffia palm fruit shells at the given optimum factor conditions of 1.06 mm, binder ratio of 29.41 % and the drying time of 3 days for the synthetic gum based briquettes and 1.99 mm particle size, binder ratio of 10 % and the drying time of 3 days for the starch-based briquettes; The relaxed density and calorific value of both

briquettes produced using synthetic gum and starch binders was evaluated experimentally and the results compared with the predicted values as earlier given by the Box-Benkhen design (BBD) in RSM (Table 1).

For the synthetic gum-based briquettes the predicted result for the relaxed density and calorific value were 451.99 kg/m³ and 21535.3 KJ/kg respectively while the experimental result for the relaxed density and calorific value were 452.07 kg/m³ and 21620.6 KJ/kg respectively. For the starch-based briquettes the predicted result for the relaxed density and calorific value were 474.01 kg/m³ and 20185.5 KJ/kg respectively while the experimental result for the relaxed density and calorific value were 470.98 kg/m³ and 20200 KJ/kg respectively.

From the results, it was observed that there were little differences between the values predicted by the models developed and the experimental values but this may be due to the negligible errors made during the experimentation; since the differences between the predicted and experimental values can be seen as not being significant. It was also observed that both the predicted and experimental results fall within the acceptable standard values. This implies that, the models developed can be relied upon for the prediction of the desired responses.

3.4 Implication of Findings

The physical characteristics of charcoal briquettes including relax density and calorific value are critical factors for deciding on the quality and commercial value of briquettes. High relax density entails the transportability of the briquettes, while a high calorific value shows the energy content of the briquettes. These characteristics are influenced by variables such as the type and ratio of the binder, the particle size of the starting material, and the extent of drying which must be carefully optimized during the briquetting process (Gebresas *et al.*, 2015). Succinctly, increase in binder to substrate ratio lead to an increase in the relax density and calorific value of the briquette, while a decrease in the particle size encourages an increase in the relax density but does not alter the calorific value of the briquette. Such increased density is attributed to the release of pore space during the binder enrichment and compacting process, especially at miniature particle size of the precursor. Similar findings were reported by Fikri and Sartika (2018).

During optimization, the goals and constraints are associated with the responses and factors respectively. In this context both calorific value and relaxed density must be maximized as is desired of any energy material, the particle size was constrained in the range of the experimental levels chosen as guided by literature evidence (Aransiola *et al.*, 2019), while the binder ratio and drying time which are costly inputs were set to be minimized at the optimum conditions. Optimum calorific values of 20185.5 - 21535.3 KJ/kg as obtained in the current work are higher than those reported in previous studies (Mopoung and Udeye, 2017). This shows that both gum and starch are excellent binders for the production of charcoal briquettes and could separately be adopted in future research based on convenience and availability. Similarly, the relaxed density of the briquettes at optimum process conditions as stated earlier (451.99 – 474.01 kg/m³) are comparable with that of wood-derived charcoal and those reported for charcoal briquettes of biomass origin (Aransiola *et al.*, 2019). These findings indicate that gum or starch-bonded charcoal briquettes at optimum preparation conditions highlighted are of commercial value as their relaxed density values provide room for its packaging, transport over long distances, storage, supply and their effective utilization by the end users. The findings of the current work are further consistent with those of Oladeji and Ekwerenmadu, (2012) and Yuliah *et al.*, (2017) in their separate works.

Table 1: Optimal Predicted and Experimental Values of Briquettes

Parameters	Synthetic Gum based Briquettes		Starch Based	d Briquettes	
	Predicted	Experimented	Predicted	Experimented	Acceptable Standard Values
Relaxed Density (kg/m³)	451.99	452.07	474.01	470.98	200-2000 (ASTM, 2004)
Calorific value (kj/kg)	21535.3	21620.6	20185.5	20200	13000 AND ABOVE (ASTM, 2004)

4 CONCLUSION

Briquettes were produced from Raffia palm fruit shells using synthetic gum and cassava starch as binders. The briquette qualities were optimized by Response Surface Methodology (RSM) using rotatable Box-Benkhen Experimental Design (BBD). The raw material was carbonized and charcoal briquette samples were made by compressing the carbonized raffia palm fruit shells in the developed briquetting machine. The process factors considered for modelling and optimization were particle size, binder-to-substrate ratio and drying time. The relaxed density and the calorific value of the briquettes were the response variables. The study shows that raffia palm fruit shells can be optimally converted into durable briquettes that can be used to provide the energy required for both domestic and industrial use in Nigeria and elsewhere.

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APPENDICES



Figure S1: Optimally Produced Charcoal Briquettes

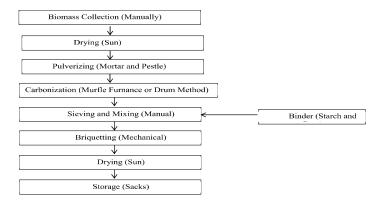


Figure S2: Process Flow Chart Diagram for Charcoal Briquette Production

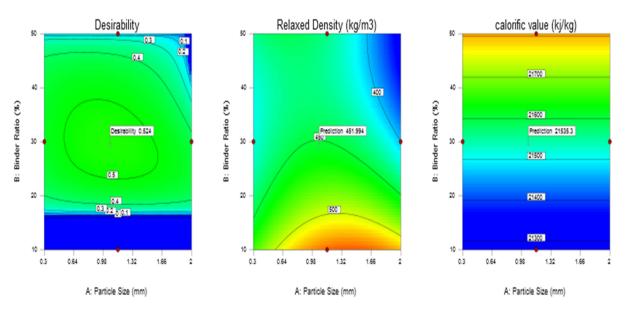


Figure S3: Response surface contour plots for optimization of synthetic gum-based briquettes

Table S1: Box-Benkhen Experimental Design for Synthetic Gum-based Briquette

Run		Parameters	Responses		
	Particle Size (mm)	Binder to substrate Ratio (%)	Drying Time (Days)	Relaxed Density (kg/m³)	Calorific Value KJ/kg
1	0.3	30	7	528.63	21468
2	2	50	5	364.98	21891
3	1.15	30	5	481.43	21468
4	2	30	7	413.56	21468
5	0.3	50	5	481.43	21468
6	2	10	5	541.03	21364
7	1.15	30	5	486.11	21468
8	0.3	10	5	526.70	21364
9	2	30	3	391.39	21468
10	1.15	30	5	481.43	21468
11	0.3	30	3	421.18	21468
12	1.15	30	5	486.11	21468
13	1.15	50	3	464.81	21891
14	1.15	10	7	520.83	21364
15	1.15	50	7	557.41	21891
16	1.15	10	3	513.70	21364
17	1.15	30	5	480.11	21468

Table S2: Box-Benkhen Experimental Design (BBD) for Briquette Produced with Cassava Starch Binder

Run		Parameters	Responses		
	Particle Size (mm)	Binder to substrate Ratio (%)	Drying Time (Days)	Relaxed Density (kg/m³)	Calorific Value KJ/kg
1	0.3	30	7	654.13	19987
2	2	50	5	411.81	19778
3	1.15	30	5	521.22	19987
4	2	30	7	375.23	19987
5	0.3	50	5	578.87	19882
6	2	10	5	458.72	20200
7	1.15	30	5	521.22	19987
8	0.3	10	5	420.42	20200
9	2	30	3	382.93	19987
10	1.15	30	5	521.22	20096
11	0.3	30	3	458.98	19987
12	1.15	30	5	521.22	19987
13	1.15	50	3	452.08	19778
14	1.15	10	7	436.30	20200
15	1.15	50	7	578.03	19778
16	1.15	10	3	415.90	20200
17	1.15	30	5	521.22	19987