Optimising Densification Condition of Coffee Husks Briquettes Using Response Surface Methodology

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ABSTRACT
This study was carried out to establish the optimum conditions for converting coffee husks with molasses as binder into a densified biomass fuel. The study was conducted following the response surface methodology (RSM) using rotatable central composite experimental design (CCD). Briquette samples were made by compressing the coffee husks-molasses mixture in a piston and die assembly. The factors in the study were the quantity of binder, die pressure, moisture content, material particle size and dwell time. The density, durability and stability of the densified coffee husks were the response variables. The results of the study indicated that a combination of die pressure of 14.91MPa, moisture content of 8.00% wb, and binder content of 45.00% wb was optimum. Under the optimum settings of the variables, the briquettes produced had a particle density of 718.09 kg/m^3, durability of 80.77% and stability of 14.98%. The study shows that coffee husks can be converted into durable briquettes that can be used to provide the energy required for the development of industry in Uganda.

Keywords: Biomass, Briquettes, Densification, Optimisation

1.0 INTRODUCTION
It is estimated 280,000 tonnes of coffee husks are generated from coffee hullers in Uganda annually (Da Silva *et. al*., 2003). However, the use of coffee husks as a domestic or industrial fuel is still very limited because in loose form, it burns with low thermal efficiency and increased air pollution. Loose coffee husk is also bulky and poses difficulty during handling, transportation and storage. Biomass densification, which is the application of mechanical pressure to loose biomass to convert it into a high density solid material, is employed to overcome these limitations (Munoz-Hernandez *et. al*., 2004).

The challenge with biomass densification is ensuring that the densified product is of high particle density, durability and stability (Mani *et. al*., 2006). These quality parameters are affected by feed material composition and the process conditions. Feed material constituents that affect the quality of densified biomass include the percentage composition of starch, protein, fibre, fat and lignin in the material (Kaliyan and Morey, 2009; Samson *et. al*., 2005). The process variables include the moisture content of the material, die pressure, binder content, dwell time, material particle size, and the temperature of the material and the die (Bhattacharya *et. al*., 1989; Samson *et. al*., 2005). The feed material composition is inherent to the material and therefore difficult to manipulate. However, the process variables can be easily manipulated in order to produce briquettes of desirable physical qualities.

The effects of the process variables on the quality of densified biomass is widely reported in literature (Samson *et. al*., 2005; Andrejko and Grochowizc, 2007; Mani *et. al*., 2006; Al-Widyan *et. al*., 2002; and Mani *et. al*. 2004). As reported by Womukonya and Jenkins (1995), the biomass densification can be carried out with binders that act as glue to hold the particles together. Alternatively, densification can be carried out without binders at elevated temperatures and pressures. For developing countries like Uganda, low pressure densification is preferred because simple manual machines can be fabricated for making briquettes, and the rate of wear of parts of the machines is reduced.

However, biomass densification is an intricate process, with each biomass material having a unique set of process variables under which to produces briquettes of desirable physical properties. Currently, the levels of factors under which to produce briquettes of optimum physical properties from coffee husks-molasses mixtures are not known. Therefore, this study was carried out to determine the levels of process variables under which to produce coffee husk briquettes of optimum quality at the lowest pressure possible, with molasses as binder.

2.0 MATERIALS AND METHODS

2.1 Preparation of samples

Coffee husks samples were collected and dried in a Solar Dryer to a moisture content of 7.20% wb. The husks were then commutated using the feed mill Pulverator® Hammer Mill, Model P-195 made by Jacobson Machine Works, Minneapolis, Minnesota (USA) with a screen size of 2.00mm fitted. The milled husk was graded to different particle sizes by screening through mesh of varying sizes. Moisture content of the graded coffee husk was regulated by adding known quantities of distilled water to the samples followed by mixing and storing in air-tight bags for 48 hours to allow for uniform distribution of the moisture, as suggested by Al-Widyan *et al*., (2002). Different proportions of sugarcane molasses were mixed with coffee husks. The mixture was placed in a mixing bowl and mixed to give a consistent material for compression into briquettes.

2.2 Method for compression of samples

The briquette samples were made by compressing the coffee husk-molasses mixture in a piston and die assembly. The design of the briquetting assembly was adapted from Andrejko and Grochowicz (2007). Briquette samples were made by placing approximately 10.00g of the sample in the cylinder with the base plate in place. This was followed by compression to a pre-determined pressure. After compression, the base plate was removed and replaced with a mandrel and the sample pushed out of the cylinder. Compression was carried out using a TQ SM100 Universal Material Testing Machine as shown in Figure 1. The samples were then dried in a tunnel-type solar drier at a temperature of $55^oC \pm 5^oC$ and on attainment of constant weight stored in air-tight bag for a period of two weeks before analysis of its quality.

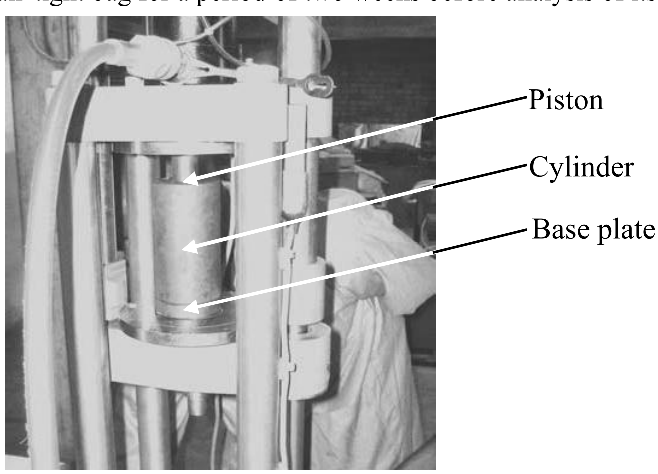

Figure 1: Compression of briquette samples using a compression testing machine

2.3 Methods for measurement of responses

The response variables for the study were durability, stability and density. Durability of the briquettes was determined according to ASAE S269.3 standard for the determination of durability. Stability of the sample briquettes were determined by expressing the change in length of the dried samples after two weeks as a percentage of the sample length immediately after ejection from the compression assembly (Wamukonya and Jenkins, 1995). Particle density was determined by dividing the mass of the sample by its volume and expressed as a percentage (Obernberger and Thek, 2004).

2.4 Optimisation procedure

Response surface methodology (RSM) procedure (Anderson and Whitcomb, 2005; Montgomery, 2005) was used for optimising the process variables. The method has been widely used in optimization studies (Chen *et. al*., 2005) because of its ability to obtain large amount of information from a small number of experiments. The method also makes it possible to observe interactions of factors on the responses making it possible to identify synergism and antagonism in a process. The first step in the RSM procedure was to screen out trivial factors of the densification process.

2.4.1 Screening of trivial factors

The standard two-level fractional factorial design was employed to eliminate the factors that did not affect the quality of the briquette. Factors that were considered during this stage of the experiment were die pressure, material moisture content, particle size, quantity of binder and dwell-time. The responses for the study were density, durability and stability of the briquettes. At every setting of the factor level, triplicate sample briquettes were made. The data collected was used to fit a first-order multiple linear regression model presented by Equation (1) for predicting the response variables (Montgomery, 2005).

$$y_i = \beta_o + \sum_{i=1}^{k} \beta_i x_i + \varepsilon \qquad (1)$$

where, y_i*'s* are the response variables while x_i*'s* are the coded factors under investigation. The term β_o is the intercept coefficient and β_i*'s* the partial regression coefficients; ε is random errors, assumed to be normally distributed with mean 0 and variance σ^2. The factor X_i was coded as x_i according to Equation (2) below (Montgomery, 2005).

$$x_i = \frac{X_i - X_o}{\Delta X_i} \qquad (2)$$

where, x_i is a dimensionless coded level of variable X_i, X_o is the real value of X_i at the centre point level and ΔX_i is the step change in the variable X_i. Coding was carried out to normalise the parameters before performing regression analysis, since the parameters have different units and ranges in the experimental domain.

2.4.2 The optimization experiment

Before carrying out optimisation experiment, region of curvature was established following the method of "path-of-steepest ascent" described by Montgomery, (2005) and Anderson and Whitcomb, (2005). Once curvature was confirmed, a rotatable central composite experimental design (CCD) (Anderson and Whitcomb, 2005; Montgomery, 2005) was employed to optimize the process variables. Second order quadratic models presented by Equation (3) were built to approximate density (y_1), durability (y_2), and stability (y_3).

$$y_i = \beta_o + \sum_{i=1}^{k} \beta_i x_i + \sum_{i=1}^{k} \beta_{ii} x_i^2 + \sum_{i=1}^{k} \sum_{j=1}^{k} \beta_{ij} x_i x_j + \varepsilon \qquad (3)$$

where, y_i are the response variables; β_o is the intercept, β_{ii} is coefficient of quadratic effect and β_{ij} is the interaction coefficient. Variables x_i and x_j are coded levels of factors X_i and X_j investigated in the experiment and ε are random errors, assumed to be normally distributed with mean 0 and variance σ^2. The regression models developed at this stage of the experiment were validated through confirmation experiments (Garg *et. al*., 2008; Anderson and Whitcomb, 2005; Montgomery, 2005).

2.4.3 Optimization methods employed

Desirability functions technique was used for optimising the densification process (Munoz-Hernandez *et. al*., 2004; Whitcomb and Anderson, 2005; Montgomery, 2005). Each response, estimated by Equation (3), was converted into individual desirability d_i that varied over the range $0 \leq d_i \leq 1$. The factor levels were chosen to maximise the overall desirability D presented by Equation (4);

$$D = (d_1 \times d_2 \times \cdots \times d_n)^{1/n} = \left(\prod_{i=1}^{n} d_i \right)^{\frac{1}{n}} \quad (4)$$

where, n is the number of response variables. Values of D close to unity imply that the factor is close to optimum (Anderson and Whitcpmb, 2005; and Montgomery, 2005). The desirability for density (d_1) and durability (d_2) were calculated using Equation (5) while that for stability (d_3) was determined using Equation (6). This desirability functions were chosen so as to maximise density and durability while minimising stability.

$$d_1 \ and \ d_2 = \begin{cases} 0 & y < T \\ \left(\dfrac{y-L}{T-L} \right)^{r_1} & L \leq y \leq U \\ \left(\dfrac{U-y}{U-T} \right)^{r_2} & T \leq y \leq T \\ 0 & y > U \end{cases} \quad (5)$$

$$d_3 = \begin{cases} 1 & y < T \\ \left(\dfrac{U-y}{U-T} \right)^{r} & T \leq y \leq U \\ 0 & y > U \end{cases} \quad (6)$$

where, T is the target value of the response y, L and U are the upper and lower limits of the desired responses. r is the desirability parameter also called weight was taken as unity for a linear desirability (Anderson and Whitcomb, 2005; Montgomery, 2005).

3. RESULTS AND DISCUSSIONS

3.1 Results of the study

From the screening experiment, dwell time was eliminated because it did not have a statistically significant (P>0.05) effect on the physical qualities of the coffee husks briquettes. Dwell time was therefore not included in the optimisation phase of the experiment. Factors that were found to be statistically significant (P<0.05) were particle size, moisture content, quantity of molasses added and die pressure applied. The factor dwell time was therefore not included in the subsequent experiments.

The result of the central composite experiment (CCD) is presented in Table 1. The bulk density of the briquettes varied between 640.40 and 723.48 kg/m^3. The highest value of durability recorded was 86.20%. Results in Table 1 were used to develop second order regression models which are given by Equations (6), (7) and (8).

Table 1: Results of the central composite design (CCD) experiment

No.	Coded Factors			Responses*		
	x1	x2	x3	Density (kg/m3)	Durability (%)	Stability (%)
1	-1	-1	-1	640.40	12.90	12.20
2	1	-1	-1	657.30	44.70	16.82
3	-1	1	-1	664.00	16.20	15.27
4	1	1	-1	682.37	18.10	19.72
5	-1	-1	1	697.41	68.40	14.01
6	1	-1	1	719.11	86.20	18.25
7	-1	1	1	692.33	47.00	14.90
8	1	1	1	698.89	46.60	18.21
9	-1.682	0	0	667.46	35.60	11.51
10	1.682	0	0	695.52	49.40	21.11
11	0	-1.682	0	702.95	52.60	14.34
12	0	1.682	0	723.48	32.90	15.25
13	0	0	-1.682	646.90	16.90	17.55
14	0	0	1.682	707.45	71.00	15.21
15	0	0	0	708.53	68.10	16.93
16	0	0	0	696.73	60.20	14.29
17	0	0	0	700.86	58.00	15.45
18	0	0	0	702.58	66.20	14.92
19	0	0	0	694.31	66.50	16.05
20	0	0	0	709.46	64.30	16.16

$$y_1 = 702.36 + 8.11x_1 + 4.24x_2 + 19.44x_3 - 9.07x_1^2 + 2.15x_2^2 - 10.59x_3^2 - 1.71x_1x_2 - 0.88x_1x_3 - 9.25x_2x_3 \quad (6)$$

$$y_2 = 63.83 + 5.44x_1 - 8.60x_2 + 18.11x_3 - 7.41x_1^2 - 7.32x_2^2 - 6.90x_3^2 - 6.01x_1x_2 - 2.04x_1x_3 - 4.71x_2x_3 \quad (7)$$

$$y_3 = 15.86 + 2.47x_1 + 0.68x_2 - 0.121x_3 \quad (8)$$

Results of confirmation experiments indicated that there is no significant difference ($p>0.05$) between the values predicted by the models developed and the experimental values. The factors that affected density significantly (at $p<0.05$) were found to be die pressure x_1, binder content x_3, the quadratic terms; die pressure x_1^2, and binder content x_3^2 and the interaction between moisture content and quantity of binder x_2x_3. The quantity of binder was found to be the most significant factors affecting the density of the briquettes followed by die pressure.

The lowest value of durability observed was 12.90% with all the three factors at their lowest levels. As indicated by Table 1, all the linear and quadratic model terms were found to have highly significantly (at $p<0.01$) effect on durability. Only the interaction term between die pressure and binder content x_1x_3; was found not to have significant effect ($p>0.05$) on the durability of coffee husks briquettes.

Results of the stepwise regression of the density, durability and stability models are given in Table 2. The stepwise regression indicates that all the models developed have a significant lack of fit ($p>0.05$) implying that the responses are not linear in the region of experimentation.

Table 2: Results from the stepwise regression results of the central composite design

Coefficient	Predicted values of model coefficients			Factor
	Density (y1)	Durability (y2)	Stability (y3)	
β_o	702.36	63.83	15.86	
β_1	8.11***	5.44***	-2.47***	x_1
β_2	4.24^{ns}	-8.60***	0.68**	x_2
β_3	19.44***	18.11***	-0.12^{ns}	x_3
β_{11}	-9.07***	-7.41***	-	x_1^2
β_{22}	2.15^{ns}	-7.23***	-	x_2^2
β_{33}	-10.59***	-6.09***	-	x_3^2
β_{12}	-1.71^{ns}	-6.01***	-	x_1x_2
β_{13}	-0.88^{ns}	-2.04^{ns}	-	x_1x_3
β_{23}	-9.25***	-4.71**	-	x_2x_3
Model (F-value)	21.64^a	40.57a	23.28^a	
R^2	0.9072	0.9733	0.8136	
Adjusted R^2	0.7361	0.9494	0.7787	
Lack of fit (p-value)	0.2869^b	0.2507^b	0.3054^b	

x_1, x_2 and x_3 are coded levels of die pressure, moisture content and quantity of binder respectively; ^{a} Significant at $p<0.0001$ level; ^{ns} Not significant

^{b} The model selected should have a non-significant lack of fit vlaue ($p>0.05$)

* Significant at $p<0.1$; ** Significant at $p<0.05$; *** Significant at $p<0.01$

3.2 Response surface plots

To visualize the combined effects of two factors on the density and durability of the briquettes, the response surface plots (Anderson and Whitcomb, 2005) were generated. The response surface plots for the interaction between die pressure and binder content on the durability and density of the briquettes are illustrated in Figure (2). The maximum briquettes density of about 712.67kg/m^3 was observed at a die pressure of about 16.50 MPa and binder content of approximately 45%. These plots indicate that durability of the coffee husks briquette increases with increase in die pressure and decrease in moisture content in the region of experimentation.

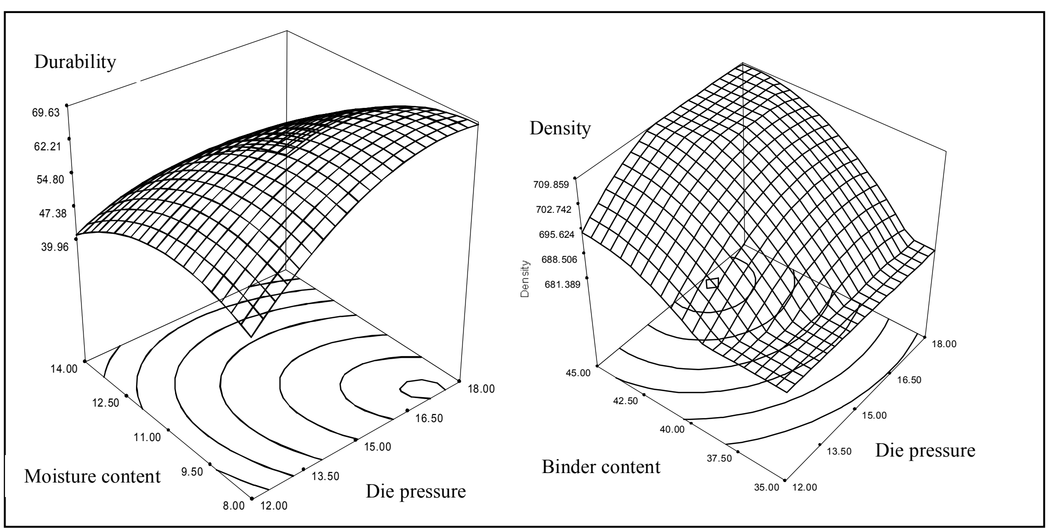

Figure 2: Response surface and contour plots of the durability response

4.5.5 Optimum conditions for densification of coffee husks briquettes

Optimum values of die pressure, moisture content, binder content were found to be 14.91MPa, 8.00%wb and of 45.00% respectively. At these factor levels the briquette produced had particle density of 718.09kg/m^3; durability of 80.77% and stability of 14.98%. The value of desirability at the optimum conditions was found to be 0.742, which is within acceptable range of 0.70 to 0.90 (Munoz-Hernandez *et. al*., 2004).

5.0 CONCLUSIONS

Die pressure, moisture content, material particle size and the quantity of binder added, are the dominant factors affecting, durability and stability of coffee husks-molasses briquettes under moderate pressures.

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