Optimization of binder addition and particle size for densification of coffee husks briquettes using response surface methodology

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Abstract. The present research focuses on establishing the optimum conditions in converting coffee husk into a densified biomass fuel using starch as a binding agent. A Response Surface Methodology (RSM) approach using Box-Behnken experimental design with three levels (-1, 0, and +1) was employed to obtain the optimum level for each parameter. The briquettes wereproduced by compressing the mixture of coffee husk-starch in a piston and die assembly with the pressure of 2000 psi. Furthermore, starch percentage, pyrolysis time, and particle size were the input parameters for the algorithm. Bomb calorimeter was used to determine the heating value (HHV) of the solid fuel. The result of the study indicated that a combination of 34.71 mesh particle size, 110.93 min pyrolysis time, and 8% starch concentration werethe optimum variables.The HHV and density of the fuel were up to 5644.66 calgr^{-1} and 0.7069 grcm^{-3},respectively. The study showed that further research should be conducted to improve the briquette density therefore the coffee husk could be convert into commercialsolid fuel to replace the dependent on fossil fuel.

1. Introduction
Coffee husk is abundantly available in coffee plantation, unfortunately no treatment and used of it. Coffee husk have a large contribution to the waste material in Middle Aceh, Indonesia, as it is well known for coffee-producing region in the Aceh Province. The production of coffee in Aceh generated significant quantities of coffee husk biomass as agro-residues.However, this material has a good potential to be analternativefuel raw materials. It contains high carbon which can be convert to solid energy source. Therefore, in one hand this might solve the environmental issues related to huge amount coffee husks waste produced each year, in another hand, it creates renewable energy.

Agro residues are usually burned in open area, which pollutes the environment. Moreover, the ash contains a largeportion of unburned carbon due to incomplete combustion.Using agricultural residues directly as energy source is not recommender due to some disadvantages: low bulk density, difficult to handle and the transport and storage are costly[1]. Therefore, biomass densification, which apply mechanical pressure, enhance the density of solid material. This process overcome these limitations [2].Using coffee husk as raw material for bio briquettes has been performed by [1]and [3]. According to Suarez et al., [1], it is possible to convert coffee husks into briquettes using the piston with preheated technology and it can be used for combustion purposes in bakery furnaces.

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According to Suarez et al., (2003) [1], and study of [3]which found that the briquetting preassure can increase the durability of coffee husk briquettes.Additionally, the optimum response of coffee husk biobriquettes obtained in briquetting pressure, moisture content and sugarcane molasses levelswere14.91 MPa, 8% and 45% respectively. In these conditions, the density of the briquettes obtained upto 718.09 kg / m^3, 80.77% durability and 14.98%.stability

Coffee husks biobriquettes can be produced directly from raw materials without pyrolysis process. However, this process has some disadvantages, high volatile matter in the material which produce smoke in the briquettes. Pyrolysis is one of the solution to increase the briquettes quality.Pyrolyslis eliminates unwanted material, such as moisture, volatile matter, and increase the briquette HHV.

Adapa, et al, [4] stated that the briquetting pressure significantly affected the density, durability and specific energy of bio briquettes. Dense briquettes showed high energy per unit volume of material. Hence, durability is the resistance of the briquettes to hold strain and force collisions that may occur during handling and transport the products [5]. Regarding to[6], the optimum conditions to produce solid fuels from rice straw biomass were 3002.8 psi of briquetting pressure, 34. 90 mesh particle size, and 7.775 % maximum raw material water content.

Previous studies about biomass pyrolysis focused on the parameter of optimization of biomass pyrolysis using response surface methodology [6, 8, 9]. Briquetting pressure on this research was relatively low (2000 psi) so that can be easily applied by society. Sustainability-income research will certainly provide a very promising prospect for the community in the countryside.

This research aimed to study the impact of particle size of coffee husk charcoal and binder concentrations on the briquettes quality. Response surface methodology was applied in order to optimize the interaction between the parameters. This result would be on behalf of promoting clean energy applications in the future.

2. Materials and Methods

The coffee husk biomass was collected from coffee hullers in the rural areas of Gayo Highlands, Aceh, Indonesia. The biomass was dried in the open air for a week, then pyrolysed in the furnace into three pyrolysis time categories, 50,100, and 150 minutes. The charcoal then grounded in a milling machine and screened to 20, 40, and 60 mesh particle size. The concentration of 8, 10, and 12 % (w/w) of starch binder, were applied in the densification process.

Gross calorific value was measured using adiabatic bomb calorimeter according to ASTM standard D- 5685. The independent variables were pyrolysis time X_1, particle size X_2, and starch binder concentration X_3 with a 2000 psi briquetting pressure as a constant variable. The dependent variables were density and heating value of briquettes. The densification experiments were conducted using bench type manually operated, laboratory hydraulic press with 20 ton (Hydraulic press Shop CMC ISO9002) capacity and a densification die. The densification die was constructed from stainless steel cylinder of 30 mm in internal diameter and 250 mm in length, equipped with stamp of 30 mm in external diameter.

A Bob-Behnken technique in RSM with three levels (low, medium and high) being coded as -1, 0, and +1 and a total of 17 runs were carried out in repetitious to optimize the level of chosen variables, pyrolysis time (min), particle size (mesh) and starch concentration (%). The test carried out according to [6], the range and the level used in the experiments are select and listed in Table 1.

Table 1. Experimental range and levels of independent variables.

Independent variable	Coded level and range		
	-1	**0**	**+1**
Pyrolysis time, X_1(min)	50	100	150
Particle size, X_2 (mesh)	20	40	60
Starch concentration, X_3 (%)	8	10	12

The density of the briquettes was measured by using the ratio of weight and volume determined from the briquette geometric shape. The research carried out by conducting the parameters from the Box-Behnken Design Experiment where consisted of 17 treatments randomly (Table 2). Tabel 2 presented the actual and prediction of heating value and density from the experiment. A total of 17 experiments had to be conducted to determine 10 coefficients of second order polynomial reationship, which modeled according to Equation (1).

$$Y_k = \beta_o + \sum_{i=1}^{3} \beta_i X_i + \sum_{i=1}^{3} \beta_{ii} X_i^2 + \sum \sum_{i<j} \beta_{ij} X_i X_j + \varepsilon_j \qquad (1)$$

In the experimental design model, Y is the predicted response and pyrolysis time, X_1 (min), particle size, X_2 (mesh), and starch, X_3 (%) were taken as independent variables. Constants β_o, β_i and β_{ij} are linier term, quadratic term and cross product term coefficients, respectively.

Table 2. Bob-Behnken Design matrix along with experimental and predicted results.

No.	Pyrolysis time, X_1(min)	Particle size, X_2 (mesh)	Starch conc, X_3 (%)	Density (gcm^{-3}) Exp.	Predicted	Heating value (calg^{-1}) Exp	Predicted
1	100	60	12	0.8204	0.7870	5433.27	5485.16
2	50	40	8	0.6565	0.6463	5238.37	5246.88
3	100	20	8	0.6262	0.6286	5711.04	5659.15
4	150	40	12	0.7288	0.7510	5503.97	5495.46
5	150	60	10	0.8011	0.8092	5282.79	5239.41
6	150	40	8	0.7680	0.7799	5548.15	5496,35
7	100	40	10	0.7402	0.7225	5665.90	5583.83
8	100	40	10	0.7063	0.7225	5539.55	5583.83
9	100	60	8	0.8208	0.7976	5323.16	5418.34
10	150	20	10	0.7392	0.7217	5552.21	5655.90
11	50	60	10	0.7452	0.7755	5337.01	5233.32
12	100	20	12	0.6846	0.6768	5634.61	5539.43
13	50	40	12	0.7129	0.7129	5143.07	5194.87
14	100	40	10	0.7016	0.7225	5523.31	5583.83
15	50	20	10	0.5790	0.5837	5068.55	5111.93
16	100	40	10	0.7286	0.7225	5634.61	5583.83
17	100	40	10	0.7233	0.7225	5555.79	5583.83

The individual desirability functions (DF) is usually performed as the optimum criteria for the purpose of optimizing multiple response variable. The Desirability Functions (DF) approach has been successfully applied for optimizing process variables for densification of rice straw as a rural alternative solid fuel [6]. Such an operating approach would be adopted in this research to get maximum or minimum value of the variable response for technical and economical considerations.

3. Results and Discussion

3.1 Calorific analysis of coffee husk biobriquettes

Direct combustion of biomass is not preferable due to the negative aspects coming from the intrinsic properties of biomass such as low density, low calorific value in a unit volume, and high moisture [10].

Therefore, it is important to develop strategies where biomass is converted to secondary fuels, which have better characteristics in comparison to the parent material[11]. For this purpose, biomass is firstly carbonize to eliminate the moisture and volatile matter contents and then the volatile matter-free solid char, which is called as "smokeless fuel", is briquetted to form firm bio-fuel briquettes.

Table 3. Calorific analysis of coffee husk.

Parameter	Heating value (calg^{-1})
• Coffee husk before carbonization (original sample)	3971.052
• Coffee husks after carbonization	5713.672

Table 3 showed the calorific analysis of the coffee husk before and after carbonization. The calorific value of the coffee husk briquettes was 5713,672 calg^{-1}. It was higher than the original sample 3971,052 calg^{-1}. According to these result it is clear that the coffee husks have a typical biomass structure with high heating value after carbonization process.

The briquetting of a smokeless fuel that obtained from the carbonization of coffee husk at 350ºC gave very good results. It contained high calorific value and almost no volatile matter content. The coffee husk briquettes is a solid fuel geen energy.

3.2 Regression Model and ANOVA for heating value and density of biobriquettes

The results of the each experiment performed was given in Table 2. Empirical relationship between the response and the independent variables have been expressed by the quadratic model Equation (2) and Equation (3).

$$Y_{1k} = 4358,199 + 24,48398\ X_1 + 8,75582\ X_2 - 40,8725\ X_3 - 0,088164\ X_1^2 \\ - 0,1332\ X_2^2 - 1,25775\ X_3^2 - 0,13447\ X_1.X_2 + 0,1278\ X_1.X_3 + 1,16588\ X_2.X_3 \quad (2)$$

$$Y_{2k} = - 0,040288 + 4,29175 \times 10^{-3}\ X_1 + 9,77312 \times 10^{-3}\ X_2 + 0,0433\ X_3 \\ - 2,6075 \times 10^{-5}\ X_1.X_2 - 2,39 \times 10^{-4}X_1.X_3 - 3,675 \times 10^{-4}\ X_2.X_3 \quad (3)$$

The response plots and a mathematical model were generated for the process. The results were determined in the correlation to coefficient of determination (R2) in the model.

Table 4. The Model fit Summary for (a) Heating Value and (b) Density

(a) Heating Value

Model Summary Statistics

Source	Std. Dev.	R-Squared	Adjusted R-Squared	Predicted R-Squared	PRESS	
Linear	170.01	0.3431	0.1915	-0.2036	6.884E+005	
2FI	171.48	0.4859	0.1774	-0.9284	1.103E+006	
Quadratic	100.00	0.8776	0.7203	-0.5613	8.930E+005	Suggested
Cubic	62.70	0.9725	0.8900		+	Aliased

(b) Density

Model Summary Statistics

Source	Std. Dev.	R-Squared	Adjusted R-Squared	Predicted R-Squared	PRESS	
Linear	0.029	0.8360	0.7981	0.6722	0.021	
2FI	0.022	0.9261	0.8817	0.6848	0.021	Suggested
Quadratic	0.022	0.9460	0.8765	0.3615	0.042	
Cubic	0.016	0.9844	0.9374		+	Aliased

The model fit summary was in Table 4. According to heating value, the quadratic model suggested model with adjusted R^2 of 0. 8776 and predicted R^2 of -0.5613. This shows that adding the quadratic (squared) terms to the mean, block, linier and the two factor interaction terms in the model is significant. Additionally, the model fit summary in Table 4 for density shows that two Factor Interaction (2FI) is selected with adjusted R^2 of 0.8817 and predicted R^2 of 0.6848. The correlation coefficients might have resulted from the insignificant terms in Table 5. It is most likely due to three different variables selected in wide ranges with a limited number of experiments as well as the nonlinier influence of the investigated parameter on the response process.

For each source of terms as depicted in Table 5, the probability ("PROB> F") was examined to see if it falls below 0.05. So far, the quadratic model and Two Factor Interaction model looks the best. These terms are significant, but adding the cubic other terms will not significantly improve the fit since it is aliased.

The SS for lack and fit is the difference between the residual SS for the simple linier regression model and the residual SS. The word "lack of fit" refers to the fact that the simple linier regression model may not adequately fit the data. If the SS for lack of fit is small there is evidence of simple linier regression model is more appropriate to explain the relationship of the parameter on coffee husk biobriqettes.

Analysis of variance (ANOVA) used to evaluate the effect linearity, quadratic, or the interaction of independent variables (factors) on the dependent variable (response). Analysis of variance for heating value and density can be seen in Table 4 and Table 5. As indicated by Table 4 and Table 5, all the linearity and quadratic model terms found to be significantly affect on heating value and density of coffee husk briquettes. In addition, interaction of pyrolysis time with particle size also have significant effect ($p > 0.05$) on the heating value and density.

Table 5. Analysis of variance (ANOVA) to describe the heating value briquettes using starch as binding agent.

Variation		Sum of Squares	Mean Square	F, Value	Prob> F	Judgement
Model		5.020E+5	55773.61	5.58	0.0169	Significant
	A	1.513E+5	1.513E+5	15.13	0.0060	Significant
	B	3539.05	43539,05	4.35	0.0754	
	C	1399.20	1399.20	0.14	0.7194	
	A^2	2.046E+5	2.046E+6	20.45	0.0027	Significant
	B^2	11953.12	11953.12	1.20	0.3105	
	C^2	106.57	106.57	0.011	0.9207	
	AB	72328.72	72328.72	7.23	0.0311	Significant
	AC	653.31	653.31	0.065	0.8056	
	BC	8699.29	8699.29	0.87	0.3820	
Residual		70002.59	10000.37			
Lack of Fit		54278.87	18092.96	4.6	0.0872	**Not significant**
Pure Error		15723.72	3930.93			
Cor Total		5,720E+005				

Quadratic models
$R^2 = 0.8776$; adj. $R^2 = 0.7203$; Std. Dev. = 100; C.V. = 1.83

Table 6. Analysis of variance (ANOVA) to describe the density briquettes using starch as binding agent.

| Variation		Sum of Squares	Mean Square	F, Value	Prob> F	Judgement
Model		0.060	0.010	20.88	<0.0001	Significant
	A	0.015	0.015	30.63	0.0002	Significant
	B	0.039	0.039	80.97	<0.0001	Significant
	C	7.069E–4	7.069E–4	1.47	0.2535	
	AB	2.720E–3	2.720E–3	5.65	0.0388	Significant
	AC	2.285E–3	2.285E–3	4.74	0.0544	
	BC	8.644E–4	8.644E–4	1.80	0.2100	
Residual		4.815E–3	4.815E–3			
Lack of Fit		3.796E–3	6.327E–4	2.48	0.1989	**Not significant**
Pure Error		1.019E–3	2.5484			
Cor Total		0.065				

Model Two-Factor Interaction (2FI)
$R^2 = 0.9261$; adj. $R^2 = 0.8817$; Std. Dev. = 0.022; C.V. = 3.04

Values of "Prob > F" less than 0.0500 indicated the model terms are significant. In the heating value case A, A^2 and AB are significant model terms. Whereas, in the density case A, B and AB are significant model terms. When the value was greater than 0.100 , it indicated the model terms are not significant.
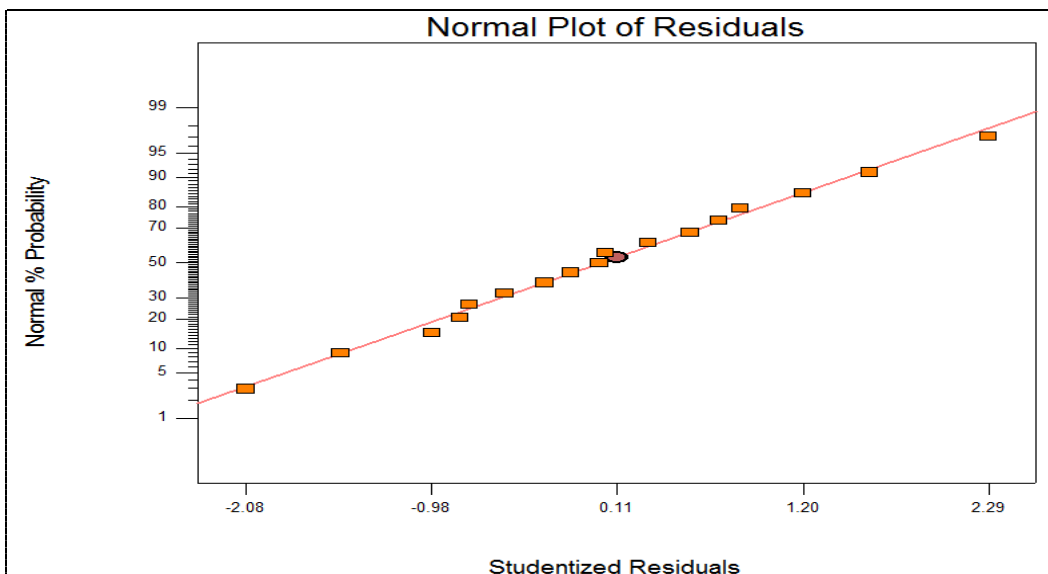
The important diagnostic tool shown in Figure 1 is the normal probability plot of the studied residuals. The data points from the plot is approximately linier, which shows that the quadratic model

developed for heating values and the two factor interaction developed for density are a good representation on the process.

(a)
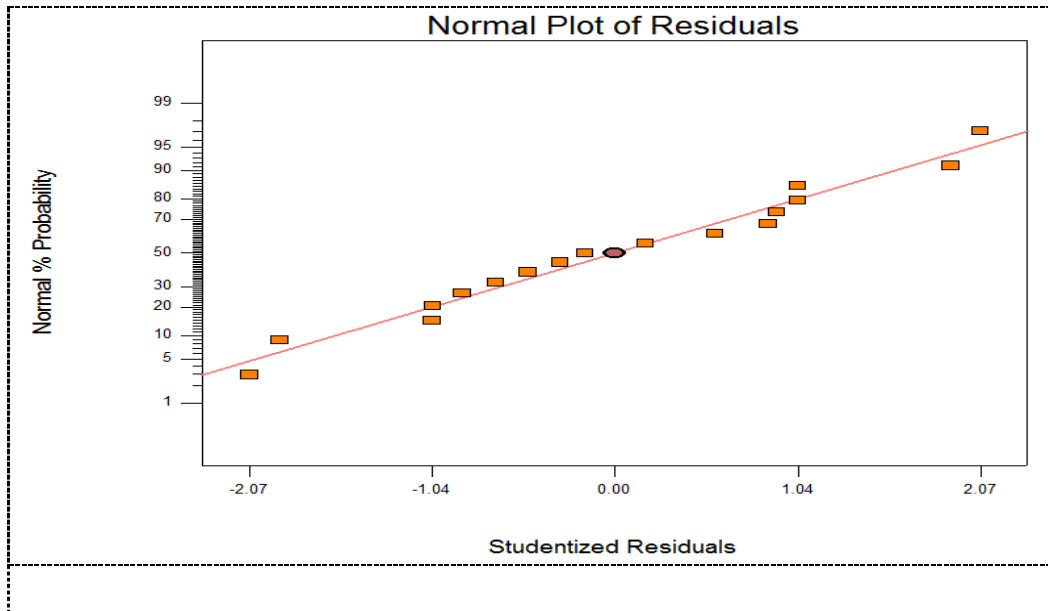
(b)

Figure 1. Normal probability plot of residuals for (a) heating value, (b) density or coffee husk biobriquettes.

3.3 Estimation of quantitative Effects of the Factors
The three dimensional response surface and contour plot obtained from second polynomial equations are shown in Figures 2-3. The values of the variable obtained as the response is optimized using the RSM technique. The best response range obtained by analyzing the response surface plots. The combined effect of particle size of coffee husk biobriquettes and pyrolysis time and binding agent 10 % on heating value is shown in Figure 2. The heating value increased with increasing the particle size of coffee husk biobriquettes. In addition, the heating value increases with the longer pyrolysis time of

raw coffee husk from 50 min to 150 min. It is noted that the maximum heating value at all the particle size achieved at 110 minute pyrolysis time of raw coffee husk.
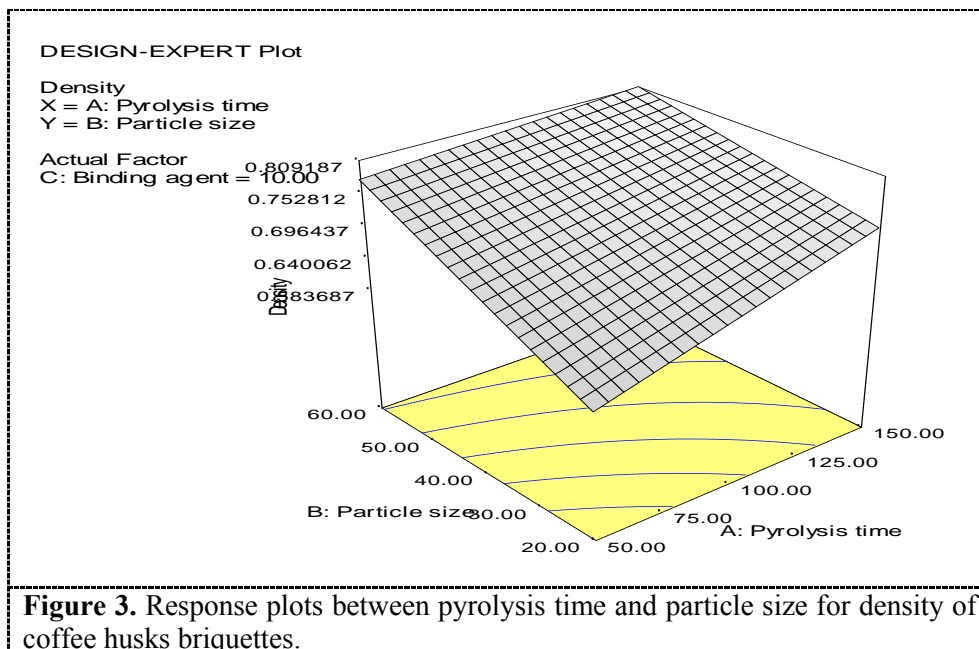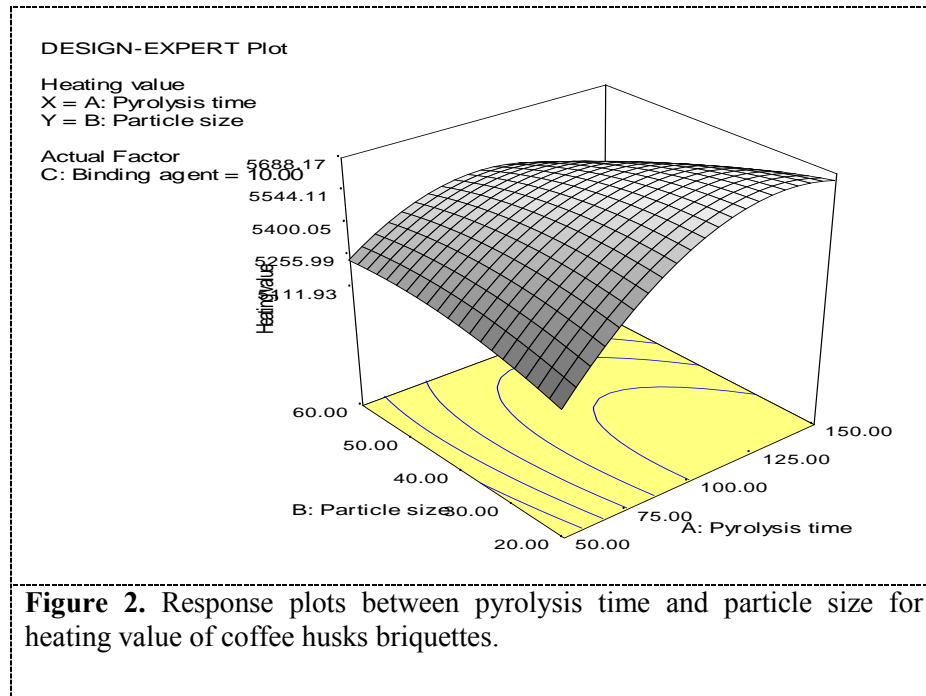

Figure 2. Response plots between pyrolysis time and particle size for heating value of coffee husks briquettes.

Figure 3. Response plots between pyrolysis time and particle size for density of coffee husks briquettes.

Figure 3 shows three dimensional response srfaces for the interaction effect of pyrolysis time and particle size on density at using 10% starch concentration. Increasing the pyrolysis time from 50 min to 150 min produced more dense biobriquettes. Consequently, the density increases with the increasing particle size from 20 mesh to 60 mesh.

3.4 Optimization of Variable Response

The optimization process aimed to get the technical parameters of the conditions that can provide optimum response with consider to economical operating cost. The quality of solid fuels was determined by its calorific value (at least 21000 J / g, SNI. No. 1/6235/2000). In order to reduce the cost of operation, the pyrolysis time and particle size should be keep to a minimum, because the longer pyrolysis process taking place and the smaller particle size, the greater energy and cost would be required. Therefore, to get the optimum response at low cost, the set-up goal pyrolysis time should be set at the "minimum" with a lower limit of 50 min and an upper limit of 150 min. For more details, refer to the table 7.

Table 7. Optimization of dependent variable of coffee husk briquettes.

No	Parameters			Response	
	Pyrolysis time (min)	Particle size (mesh)	Starch content (%)	Heating Value $(Calgr^{-1})$	Density $(grcm^{-3})$
1	82.93	57.82	8.00	5415.06	0.7735
2	83.20	57.59	8.00	5417.83	0.7747
3	84.46	55.66	8.00	5437.76	0.7648
4	88.58	54.85	8.00	5456.08	0.7650
5	110.93	34.71	8.00	5644.66	0.7069

In applying the desirability function (DF) method, the Design Expert software produced 5 solutions. Each has a DF = 1, as shown in Table 7. As the heating value of the fifth solution is the highest among the available solutions, as well as particle size, this yields the lowest density of the product. Although the fifth solution produce the longest pyrolysis time, it gives the lowest operational cost. It is therefore appropriate to select the fifth acceptable solution as the optimum. The optimum conditions to produce solid fuels from coffee husk biomass were obtained at 110.93 min pyrolysis time particle size of 34.71 mesh and starch concentration of 8%. The heating value obtained up to 5644.66 $calg^{-1}$ and density about 0.77354 gcm^{-3}.

4. Conclusions

A desirability function approach has been used to optimize the process variables of pyrolysis time, particle size, and starch content on the multiple-response variables of density and heating value of coffee husk briquettes produced through a mechanical densification. The optimum conditions to produce solid fuels from coffee husks biomass were obtained at a pyrolysis time of 110.93 min, particle size of 34.71 mesh and starch content of 8%. The heating value and density of briquette were accordingly 5644.66 $calg^{-1}$ and 0.77354 gcm^{-3}.

Acknowledgements

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