

Optimization of Particle Size and Addition of Vinasse Waste to Improve Characteristics of Rice Husk Charcoal Briquettes

Optimasi Ukuran Partikel dan Penambahan Limbah Vinasse dalam Meningkatkan Karakteristik Briket Sekam Padi

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Abstract

Biobriquettes are a biomass fuel with high calorific value. This study aims to determine the optimal conditions for the particle size of husk charcoal and the addition of vinasse waste as an independent variable, using the Response Surface Methodology and the Central Composite Design (CCD) method on Design Expert 13 software. The sample mixed with tapioca starch (8:1), which acts as an adhesive, and add vinasse waste in 3, 6, 9, and 12 mL amounts. Carbonization process, which is then mashed and sieved according to particle sizes of 20, 30, 40, 50, and 60 mesh. Then, the sample is mixed with tapioca starch as adhesive with the ratio of charcoal and adhesive 8:1 as well as vinasse waste in volumes of 3, 6, 9, and 12 mL. The resulting briquette samples were tested in the form of water content, ash content, and calorific value tests. The optimum conditions that have a significant effect on the response variable are the combination of particle size variables of 35.152 mesh and the volume of vinasse waste of 6.049 mL. The moisture content obtained was 6.696%, The ash content was 5.450%, and the calorific value was 5003.399 cal/g with a desirability value of 0.927 in the quadratic model.

Keywords: biobriquettes; optimization; Response Surface Methodology; rice husk briquettes

Abstrak

Biobriket merupakan salah satu bahan bakar biomassa yang memiliki nilai kalor yang tinggi. Penelitian ini bertujuan untuk mengetahui kondisi optimal pada ukuran partikel arang sekam dan penambahan limbah vinasse sebagai variabel bebas dengan menggunakan Response Surface Methodology metode Central Composite Design (CCD) pada perangkat lunak Design Expert 13. Proses pembuatan briket arang sekam padi dilakukan dengan proses karbonisasi yang kemudian dihaluskan dan diayak sesuai dengan ukuran partikel yaitu 20, 30, 40, 50, dan 60 mesh. Kemudian, sampel dicampur dengan tepung tapioka sebagai perekat dengan perbandingan arang dan perekat 8:1 serta limbah vinasse dengan volume 3, 6, 9, dan 12 mL. Sampel briket yang dihasilkan dilakukan pengujian berupa uji kadar air, kadar abu, dan nilai kalor. Kondisi optimum yang berpengaruh signifikan terhadap variabel respon adalah kombinasi variabel ukuran partikel 35,152 mesh dan volume limbah vinasse 6,049 mL. Kadar air yang diperoleh sebesar 6,696%, kadar abu sebesar 5,450%, dan nilai kalor sebesar 5003,399 kal/g dengan nilai desirability sebesar 0,927 pada model kuadratik.

Kata kunci: biobriket; briket sekam padi; optimasi; Response Surface Methodology

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1. Introduction

With the growing times, alternative gas fuels are needed to reduce the increasing consumption of LPG. One alternative that can be used is the utilization of biomass as a more environmentally friendly fuel with raw materials that generally come from industrial and agricultural waste.

Charcoal briquettes, a product derived from biomass utilization, represent an alternative fuel that is both easy to produce and implement [1]. They are made by mixing fibrous waste with adhesives to bind charcoal particles together, ensuring the briquettes are durable and resistant to breakage [2]. In the process, low-density biomass is processed into a high-density, energy-concentrated fuel [3]. The biomass feedstock is selected based on its high content of the main organic compound, cellulose. Rice husk is often utilized as a fuel because rice husk is composed of cellulose fibrous tissue that contains a lot of very hard silica [4]. Generally, rice husk contains about 31% cellulose and 16% silica [5]. The quality of the briquettes depends on the physicochemical properties of the biomass (particle size and surface area), as well as parameters such as compression rate, residence time, temperature, pressure, and the adhesive used [6].

Vinasse is the liquid waste that remains after the ethanol distillation process. Vinasse can be used to produce organic adhesives (lignosulfonates) which have many applications in various industries [7]. Vinasse also has a high content of salts, organic compounds, and inorganic compounds such as carbohydrates, proteins, nitrogen, sulfur, minerals, phosphorus, and other heavy metals. Vinasse has 3.1% carbon. Due to

their, carbon content, vinasse can be applied as an ingredient in the manufacture of briquettes because they can improve physical properties, be able to accelerate the ignition of briquettes, and produce a high enough briquette combustion temperature [8].

Briquettes have several quality parameters including moisture content, ash content, and calorific value. High moisture content in briquettes reduce their quality, a problem exacerbated by smaller charcoal particle sizes, which tend to retain moisture due to smaller pore sizes, making drying more difficult. According to SNI 01-6235-2000, the standard moisture content of briquettes should not exceed 8%. Lower ash content indicates higher-quality briquettes, as they burn more efficiently without turning into ash during combustion. According to SNI 01-6235-2000, the maximum ash content is 8% [9]. Calorific value is another crucial indicator of briquette quality, influenced by moisture content, ash content, carbon content, and volatile matter. According to SNI 01-6235-2000, the minimum calorific value of briquettes is 5000 calories per gram.

In previous studies, experience may decrease in quality, but they can evenly distribute heat, ensuring complete combustion [10]. However, there has been limited research on optimizing the addition of vinasse waste in order to improve the quality of briquettes. Optimization in the research can be achieved through the Response Surface Methodology (RSM) method, an experimental approach necessary to understand how various factors affect the response. The aim is to identify the conditions that yield the optimal response.

Experimental designs include Full Factorial 3-Level Design (FFD), Box-

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Behnken Design (BBD), and Central Composite Design (CCD). CCD in the optimization process to determine the approximate optimal conditions with unknown optimization and optimal location. In CCD, the test points are taken based on the test limit values determined for each research factor. The response data obtained is modeled by the appropriate mathematical model. In CCD, there are several models, namely mean, linear, quadratic, 2-factor interaction (2FI), and cubic [11].

In this study, optimization is needed on the particle size factor and the volume of vinasse waste. This is because the particle size can affect the density of the briquette. Whichever the particle size, can increase the density of the briquette. The higher density value also produced the higher heating value [12]. Then, for optimization of the volume of vinasse waste, the addition of vinasse waste concentration can affect the physical quality of briquettes because vinasse waste contains various organic and inorganic compounds that can provide more even heat and increase combustion temperature [8].

2. Research Methods

This research was conducted through several steps. Starting with the carbonization of rice husks, crushing, screening, mixing with adhesives, and printing. Then the briquette samples were tested for moisture content, ash content, and calorific value, the results of which were optimized using the Response Surface Methodology (RSM) method with Design Expert 13 software to determine the optimum particle size and addition of vinasse waste.

2.1 Tools and Materials

The tools were used in this research include a sieve, an oven, an analytical balance, pipe mold, and a furnace. The materials used in this research include rice husk, vinasse waste, and tapioca flour.

2.2 Research Procedure

The research was conducted in three steps. The first step involves the process of making briquettes, which includes carbonization, briquette formation, printing, and drying. Rice husk is carbonized in a furnace with a temperature of 250°C to obtain rice husk charcoal. The charcoal is then sieved to uniform sizes of 20, 30, 40, 50, and 60 mesh. It is mixed with tapioca glue adhesive in a ratio of 1:8 (tapioca glue to charcoal) and vinasse waste in volumes of 3, 6, 9, and 12 mL. Subsequently, the mixture is printed and dried into briquettes at a temperature of 75°C.

The second step is the process of testing the quality of briquettes, including analyzing moisture content, ash content, and calorific value. The third step is the process of processing data analysis results to obtain optimal results using RSM Design Expert 13.

2.3 Briquettes Analysis

The analysis of briquettes consisted of moisture content analysis, ash content analysis, and calorific value analysis. Moisture content shows the value of moisture content tends to increase with the smaller particle size of charcoal briquettes. This is because the smaller the particle size, the smaller the pore size produced, so that during drying, the water in the briquette will be difficult to evaporate [9]. In this research, the analysis of moisture content using ASTM (American Standard

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Testing and Material) D 5142 - 02 with the equation (1) [13].

$$\text{Moisture Content}(\%) = \frac{BD - AD}{BD} \times 100\% \dots(1)$$

Description:

BD =Weight before drying in the oven (g)

AD =Weight after drying in the oven (g)

Ash content greatly affects the heating value. A low ash content can produce a high heating value. High ash content can reduce the calorific value of the briquettes, which makes the quality of the charcoal briquettes decrease. According to SNI, a good moisture content for briquette products is less than 8% [14]. In this study, the ash content analysis used ASTM D 5142 - 02 with the equation (2) [13].

$$\text{Ash Content}(\%) = \frac{\text{Ash weight}}{\text{Sample weight}} \times 100\% \dots(2)$$

Calorific value is the energy that is transferred through a system due to the temperature difference between the system and the environment, which is called heat [15]. The calorific value of fuel is said to be the maximum amount of heat energy released through complete combustion of the fuel itself. The calorific value of fuel can be known with a bomb calorimeter tool. The principle of calculating the total energy in briquettes is the amount of heat

produced when one briquette is completely oxidized in a bomb calorimeter. Based on SNI. 01-6235-2000, charcoal briquettes are said to be of quality if the calorific value in briquettes is more than 5000 cal/g [16].

After the analysis, the data is processed into RSM optimization. In this research, optimization is carried out using the RSM method with the CCD type whose parameters can be seen in the Table 1.

3. Results and Discussion

Optimization was carried out with the factors of particle size and volume of vinasse waste on physical properties, namely moisture content, ash content, and calorific value of rice husk charcoal briquettes, as well as responses on the dependent variable from 11 experiments using the Design Expert 13 application. Experimental design in the form of CCD can be seen in Table 2.

Table 1. Parameters of CCD for briquette making process

Level	Factor	
	A: Particle Size	B: Vinasse Waste Volume
Low (-1)	20	0
Moderate (0)	40	6
High (1)	60	12

Table 2. Data experimental with CCD optimization

Run	Particle Size (Mesh)	Vinasse Waste Volume (mL)	Moisture Content (%)	Ash Content (%)	Calorific Value (cal/g)
1	20	0	6.82	5.85	5000.32
2	20	6	6.71	6.57	4764.43
3	20	12	6.78	5.33	4962.66
4	40	0	7.45	6.75	4521.66
5	40	6	6.8	5.39	4989.07
6	40	6	6.58	5.38	4881.67
7	40	6	7.57	6.8	4654.68
8	40	12	7.98	6.89	4593.17
9	60	0	6.98	6.72	4937.95
10	60	6	6.83	5.45	5002.81
11	60	12	6.84	6.41	4845.57

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Based on the data analyzed, the briquettes with the highest moisture content were obtained in briquettes with the addition of 12 mL of vinasse waste. This occurs because vinasse waste has a high moisture content. The results of previous research shows that the moisture content in briquettes using vinasse waste is higher than briquettes not using vinasse, but the use of vinasse waste produces briquettes with stronger physical properties because they can dry optimally through drying [17].

Meanwhile, the briquettes with the highest ash content have a 40 mesh particle size variable, where the particle size affects the high ash content of the briquettes. Ash content is directly proportional to particle size, where the smaller particle size causes the briquette to have smaller gaps (pores) between its particles so that the density of the briquette will be greater and will produce more ash in the combustion process of briquette [18]. High ash content decreases the calorific value of the fuel and reduces its flammability, and it contributes to high thermal resistance, leading to reduced heat transfer into the combustion chamber [19].

In this study, the calorific value ranged from 4521.66 - 5002.81 cal/g, where the highest calorific value was found at 60 mesh particle size with the addition of 6 mL vinasse. This is in line with the more moisture content in the

briquette, the heat will first evaporate the water so that the calorific value of the briquette is low. The larger mesh size causes the briquette to have large gaps (pores) between its particles so that the briquette water will be easier to evaporate and show the value of the water content of the briquette is low and the high calorific value.

Table 3 shows the response model suggested by Design Expert 13 based on the Analysis of Variance (ANOVA) data on each parameter in the quadratic model. In the quadratic model, the Standard Dev column quadratic model is chosen because it has the smallest value among the other models. Std. Dev shows the standard deviation value, or the square root of the mean square of the error which determines the distribution of the data, so the greater the standard deviation value, the more diverse and can be said to be more inaccurate; conversely, the smaller the standard deviation, the more similar the values on the item or the more accurate [11]. In ANOVA, the quadratic model is considered appropriate if the difference between the values of Adjusted R² and Predicted R² is less than 0.2. This small difference indicates the absence of noise, leading to accurate estimates. All three responses give a difference of no more than 0.2, so the quadratic model is said to be significant in determining the three responses.

Table 3. Response mathematical model analysis

Response	Math's Model	Std. Dev	R ²	Adj. R ²	Pred. R ²	Adeq. Prec.	PRESS	F-Value	P-Value	Lack of Fit
Moisture	Quadratic	0.084	0.981	0.962	0.845	20.924	0.297	51.54	0.0003	0.052
Ash	Quadratic	0.113	0.985	0.971	0.863	18.994	0.591	66.7	0.0001	0.164
Calorific Value	Quadratic	29.56	0.986	0.971	0.871	21.127	39029.4	68.24	0.0001	0.272

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The adequate precision value is used to measure the ratio of signal to noise or interference, which shows a good response because the value obtained is greater than 4. Adequate precision compares the predicted value range at the design points to the average prediction error. Ratios greater than 4 indicate sufficient model discrimination. In this particular case, the value is well above 4 [20]. So, the quadratic model in 3 responses can be said to make sense in making predictions or further experiments.

The R^2 value indicates the proportion of data variability that has a relationship between the predicted data and the experimental data [21]. A high R^2 value (close to 1) indicates that there is a satisfactory fit between the prediction data and the experimental data. The interpretation of the R^2 value in the interval of 0.8 - 1.0 is stated to be the level of relationship between the independent variable and the dependent variable, which is very strong [22]. In the response to moisture content, the high R^2 value shows a high correlation between observation and prediction values, stating that 98.1% (0.981) of the moisture content in briquettes is influenced by the particle size factor and the volume factor of vinasse waste added in the briquetting process. While the remaining 1.9% is influenced by other factors not included in the model.

The PRESS (Predicted Error Sum of Square) value is a value that states the ability of the model to predict observations that are not used in a new experiment. The smaller PRESS value indicates that the prediction results are improving. The lowest PRESS value is shown in the quadratic model, which means that the quadratic model has the best ability to

predict the response with the lowest error rate.

In ANOVA analysis, the P-value provides an indication of the significance of a model relative to the F-value. For a given F-value, it can be defined as the probability that a variable did not affect the response. If the P value for the model is less than 0.05, the model is said to be significant, which means that there is a 5% chance that the F value is due to noise. If the P-value is above 0.1 (10%), the model is said to be insignificant [23]. The P-value of the quadratic models in each response is less than 0.05 so that the relationship between variables can be declared significant or affect each other. While the F-value in the quadratic model shows a value greater than the other models, it is also declared significant, and the lack of fit value, which is greater than 0.05 (5%) indicates inaccuracy, so it is declared insignificant. The lack of fit is insignificant, implying that the model is suitable for use. So the quadratic model has a significant effect on the 3 responses.

3.1 Moisture Content with RSM Analysis

Based on the quadratic model, the moisture content response has a significant effect because it has a P-value of 0.0003 (0.03%) not greater than 0.05, which indicates the presence of noise or disturbance of only 0.03%. While the F value of 51.54 forms a model that can state the moisture content response well, and the lack of fit value obtained is 0.0517 (5.17%) which is greater than 0.05 (5%) so the inaccuracy can be declared insignificant, implying that the model is suitable for use. This RSM model can be applied with the RSM equation or model for the moisture content response shown in equation (3).

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$$Z = 7.8717 - 0.0817x + 0.01038y - 0.000542xy + 0.001239x^2 - 0.000263y^2 \dots\dots\dots(3)$$

Description :
 Z = Moisture Content of Briquettes
 x = Particle Size
 y = Vinasse Wate Volume

The response surface shape is used to describe the effect of independent variables on the response. The quadratic model equation indicates that the regression line formed is parabolic. The response surface shows the relationship between the two independent variables and their effect on the response moisture content of rice husk charcoal briquettes, which can be seen in Figure 1.

Figure 1 shows the highest color in briquettes with a particle size of 60 mesh with or without the addition of vinasse. The larger particle size causes the briquette to have large gaps (pores) between the particles, so the density is getting low. In the drying process, the briquette water will be easier to evaporate. Figure 1 also shows the relationship between moisture content and the volume of vinasse waste added, which is directly proportional where the more vinasse waste added, the higher the moisture content of the briquette. This is because vinasse waste has a high moisture content. However, under conditions that are considered optimum, between 40 and 50 mesh particle size with the addition of vinasse, the moisture content of the briquettes does not exceed the SNI limit.

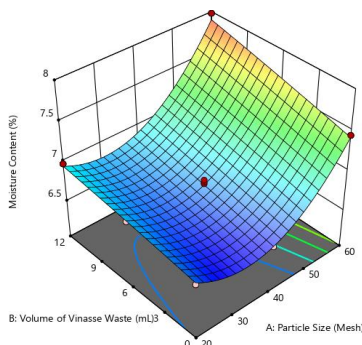


Figure 1. 3D response of moisture content

3.2 Ash Content with RSM Analysis

Based on the quadratic model, the moisture content response has a significant effect because it has a P-value of 0.0001 (0.01%) not greater than 0.05 which indicates the presence of noise or disturbance of only 0.01%. While the F-value of 66.7 forms a model that can state the moisture content response well. The lack of fit value obtained is 0.164 (16.4%), which is greater than 0.05 (5%) so the inaccuracy can be declared insignificant, implying that the model is suitable for use. The RSM model can be applied with the RSM equation or model for the moisture content response shown in equation (4).

$$Z = 9.8606 - 0.2264x - 0.00359y - 0.000021xy + 0.0029x^2 + 0.004825y^2 \dots\dots\dots(4)$$

Description :
 Z = Ash Content of Briquettes
 x = Particle Size
 y = Vinasse Wate Volume

The response surface shape is used to describe the effect of independent variables on the response. The quadratic model equation indicates that the regression line formed is parabolic. The response surface shows the relationship between two independent variables and their influence on the ash content response of rice husk charcoal briquettes, which can be seen in Figure 2.

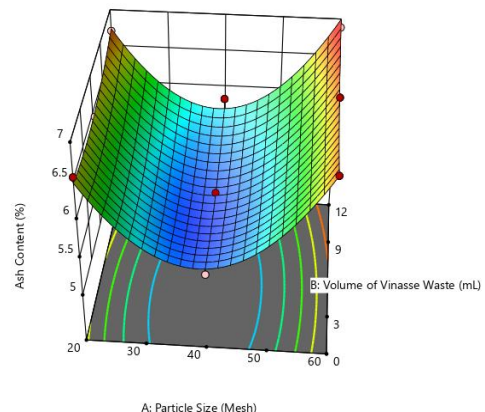


Figure 2. 3D response of ash content

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Figure 2 shows the 3D contour plot graph of ash content response with particle size and vinasse volume. It can be seen that the highest color is located in the briquette sample with a particle size of 60 mesh without the addition of vinasse. This shows that the highest ash content value is located in the briquette with a particle size of 60 mesh without the addition of vinasse. According to Thoyeb et al., ash content is directly proportional to particle size; the smaller the particle size, the higher the ash content of the briquettes [18]. The smaller particle size causes the briquette to have smaller gaps (pores) between its particles, resulting in a higher density of the briquette and more ash. In addition, briquettes with low density allow more oxygen flow in the gaps between particles so that combustion can be carried out more completely and produce less ash. Figure 2 shows the relationship between ash content and the volume of vinasse waste added, which is directly proportional, where the more vinasse waste added, the higher the ash content of the briquettes. This is because vinasse waste has a high ash content.

3.3 Calorific Value with RSM Analysis

Based on the quadratic model, the calorific value response has a significant effect because it has a P-value of 0.0001 (0.01%) not greater than 0.05, which indicates the presence of noise or disturbance of only 0.01%. While the F-value of 68.24 forms a model that can state the moisture content response well. The lack of fit value obtained is 0.272 (27.2%), which is greater than 0.05 (5%), so the inaccuracy can be declared insignificant, implying that the model is suitable for use. The RSM model can be applied with the

RSM equation, a model for the calorific value response shown in equation (5).

$$Z = 4145.1985 + 41.9399x + 33.566y - 0.2125xy - 0.5893x^2 - 1.2469y^2 \dots\dots\dots(5)$$

Description :
 Z = Calorific Value of Briquettes
 x = Particle Size
 y = Vinasse Waste Volume

The response surface shape is used to describe the effect of independent variables on the response. The quadratic model equation indicates that the regression line formed is parabolic. The response surface form shows the relationship between the two independent variables and their influence on the response of the calorific value of rice husk charcoal briquettes, which can be seen in Figure 3.

Figure 3 shows the 3D contour plot graph of the heating value response with the particle size factor and the volume of vinasse waste. It can be seen that the highest color is in briquettes with a particle size of 40 mesh with the addition of vinasse. At the condition that is considered optimum, the calorific value of the briquettes decreases, which is around the particle size of 60 mesh with the addition of vinasse. This is because the smaller particle size causes the briquette to have small gaps (pores) between the particles in the briquette so that it has a high-density value. The briquette's high density value causes its calorific value to be lower. This is consistent with the higher moisture content in the briquette. The heat will first evaporate the water, resulting in a low calorific value. In addition, briquettes with a particle size of 60 mesh have a high density, making it difficult for oxygen to be distributed in the briquettes.

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Figure 3 also shows the relationship between the heating value and the volume of vinasse waste added, which is directly proportional, where the more vinasse waste is added, the higher the heating value of the briquettes. This is because there are several contents, such as carbon and oxygen, in vinasse waste, which are quite high. However, in conditions that are considered optimum, the calorific value of the briquettes decreases, around the particle size of 60 mesh with the addition of vinasse.

3.4 Optimization of Process Conditions

Optimization in this study was carried out to determine the value of factors to produce the optimum response value. In optimization, there are several target parameters, including the upper limit, lower limit, and importance of each independent variable. This is needed to get a combination of variables that are within the upper and lower limits to achieve SNI criteria.

Table 4 shows the parameters to determine the combination that will obtain some recommended conditions, with the importance of each parameter shown. The importance value in determining the

optimum condition is needed to find out how important each variable is to get the optimum result. The importance of 3 or declared important in the particle size and volume of vinasse waste is given to provide a balanced combination between the two. The importance of 4 or very important is given to the results of the ash content and moisture content parameters given to provide the smallest results or values and not exceed the maximum value of 8. Importance 5 or very-very important is given to the results of the calorific value parameter to provide high results above 5000, where the value of importance 5 is intended because the results of the research data conducted have not fully met the standards (>5000 cal/g).

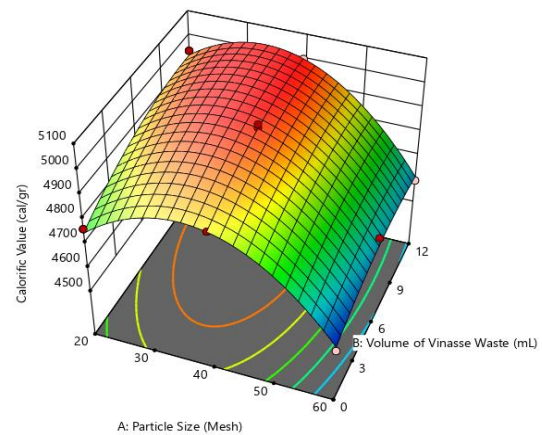


Figure 3. 3D response of calorific value

Table 4. Parameters for determining the optimum condition combination

Variable	Target	Lower Limit	Upper Limit	Importance
A : Particle Size	<i>is in range</i>	20	60	+++
B : Vinasse Waste Volume	<i>is in range</i>	0	12	+++
Moisture Content	<i>minimize</i>	6.58	8	++++
Ash Content	<i>minimize</i>	5.33	8	++++
The Calorific Value	<i>is target = 5050</i>	4521.66	5100	+++++

Description : *importance 1 (not very important); 2 (somewhat important); 3 (important); 4 (very important); 5 (very - very important)

Table 5. Optimum condition based on RSM

Particle Size (mesh)	Vinasse Waste (mL)	Moisture Content (%)	Ash Content (%)	The Calorific Value (cal/g)	Desirability
35.152	6.049	6.696	5.450	5003.399	0.927 <i>Selected</i>

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Furthermore, the optimum condition is determined based on the desirability value. Desirability states how close or fulfilled the response value is to the targeted value so that the selected optimum condition is expected to have a desirability value close to one (1.0). The desirability value becomes an optimization goal that shows the value of optimization accuracy and states the ability of the program to meet the specified parameters.

Table 5 shows the solution for the Design Expert 13 calculation system's general conditions. The optimum point with the best response results was obtained at a combination of particle size of 35.152 mesh with a volume of vinasse waste added of 6.049 mL. Based on this combination of variables, the moisture content was obtained at 6.696%, ash content at 5.45%, and calorific value at 5003.399 cal/g. In addition, the desirability value obtained is 0.927, which shows that the optimum point obtained based on the program is very good because it is close to

the value of 1. Hidayat et al., [24] shows that the desirability value of 1 indicates the perfect case. Where the desirability value is a characteristic used to explain how well the optimal solution given is by the objectives of the response.

4. Conclusion

Response Surface Methodology (RSM) provides a model to predict the response of moisture content, ash content, and heating value, namely the quadratic model. Particle size influences the results of the analysis of moisture content and ash content, and vinasse waste can increase the heating value. The results of RSM optimization provide a combination of optimal conditions from the process of making briquettes from rice husks with the addition of vinasse waste, which is located at a size of 35.152 mesh and a volume of vinasse waste of 6.049 mL, which produces a moisture content of 6.696%, an ash content of 5.45%, and 5003.399 cal/g with a desirability of 0.927.

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