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Research Article

Bio-briquettes from tea fluff biochar: a response surface methodology study on particle size, resin gum-adhesive, and used cooking oil immersion time

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Abstract. Fluff tea is the residual solid waste generated in the green tea industry and holds the potential for development as a solid fuel in bio-briquettes. This study transformed fluff tea into bio-briquettes utilizing biochar produced through slow pyrolysis. The study aimed to optimize bio-briquettes production from fluff tea using the Response Surface Methodology (RSM) approach through proximate analysis. The cylindrical bio-briquettes were produced using biochar particle sizes of 850, 500, and 150 μm , resin gum adhesive concentrations of 10%, 15%, and 20%, and immersion times in cooking oil of 0, 3, and 6 minutes. The results showed that the overall response by the p-value was <0.05 , and the lack of fit was insignificant (p-value >0.05). The findings indicated that the calorific value of tea fluff rose from 4,482.56 cal/g to 6,374.98 cal/g after conversion to biochar. The optimum conditions for producing tea fluff bio-briquettes were a particle size of 850 μm , adhesive concentration of 11%, and immersion time of 5 minutes. The bio-briquettes exhibited a moisture content of 3.53%, ash content of 5.65%, volatile matter of 14.75%, fixed carbon of 76.14%, calorific value of 7,796.37 cal/g, combustion rate of 0.11 g/min, density of 1.22 g/cm³, and compressive strength of 35.57 N/cm². Most tea fluff briquettes' properties had met Indonesia's briquettes standard. The production of bio-briquettes from tea fluff waste is a viable alternative fuel for both industrial and domestic applications.

Keywords: Bio-briquettes, Box-Behnken Design (BBD), Resin Gum Adhesive, Response Surface Methodology (RSM), Tea Fluffs, Used Cooking Oil Immersion.



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1. Introduction

The National Energy Policy in Indonesia aims for a minimum utilization of new and renewable energy at 23% by 2025 and 31% by 2050 (Sarante, 2024). Bio-briquettes, a form of renewable energy, have garnered significant interest from researchers. It can be produced from biomass, which is abundant in Indonesia. Different types of biomass have been utilized as raw materials for bio-briquettes, including rice husks, straw, coconut shells, sawdust, wood, leaves, twigs, grass, rice straw, paper, and other carbonizable agricultural waste (Akolgo *et al.*, 2021; Magnago *et al.*, 2020; Trubetskaya *et al.*, 2019). Bio-briquettes serve as a renewable alternative energy source, capable of replacing fossil fuels, including coal and petroleum.

Tea fluffs represent a byproduct of tea processing, generated from sorting twigs and leaves, accounting for 1-3% of total tea production. Tea fluffs serve as a valuable composting material due to their essential nutrients, which are crucial for the

initial growth of plants (Dewi & Wulansari, 2023). The utilization of tea fluffs as the primary component in bio-briquettes' production remains under-researched. The nutritional composition of tea fluff includes organic nitrogen (N) at 1.75-1.84% and carbon (C) ranging from 8-21%, resulting in a carbon-to-nitrogen ratio (C/N) of 10-12 (Wulansari & Rezamela, 2020).

Aini *et al.*, (2023), reported that the average carbon content of tea fluffs is 43.62%. Processing this material into biochar through pyrolysis at temperatures ranging from 300 to 500°C yielded a calorific value between 5,962.04 and 6,240.18 cal/g. Pyrolysis is a thermal decomposition process without oxygen, transforming biomass into biochar, bio-oil, and gas (Hadey *et al.*, 2022; Hu *et al.*, 2024; Iskandar & Rofiatin, 2017a; Kozlov *et al.*, 2015; Mangmang *et al.*, 2018; Martin *et al.*, 2024; Mukherjee & Lal, 2014; Susanti & Dewi, 2023; Yanmei *et al.*, 2017; Zhongyi *et al.*

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al., 2016). This process enhances the calorific value of biomass, making it suitable for applications such as activated carbon or alternative fuels like briquettes (Ridhuan *et al.*, 2018).

As an alternative energy, bio-briquettes must have a high calorific value for daily and industrial applications. The standard calorific value of briquettes in Indonesia is 5,000 cal/g, as specified in SNI 01-635-2000. However, in addition to the calorific value, the quality of briquettes is also determined by other factors such as moisture content, ash content, volatile substances, and fixed carbon. Briquettes with low moisture content can increase calorific value and combustion efficiency(Araujo *et al.*, 2016; Espuelas *et al.*, 2020; Haryanti, *et al.*, 2023; Lubwama *et al.*, 2020; Trubetskaya *et al.*, 2019). High-quality briquettes should exhibit low levels of ash and volatile content. Briquettes with low ash content generate minimal solid residue post-use, whereas volatile substances in briquettes may affect their combustion properties. High volatile content can increase smoke production; therefore, the optimal range for volatile content is between 15% and 25%. Within this range, it generates minimal smoke(Allo *et al.*, 2018). According to Yuliah *et al.* (2017), briquettes are deemed high quality when they exhibit low moisture content, ash, and volatile matter.

Fixed carbon and calorific values are also critical determinants of briquette quality. A higher fixed carbon content is associated with an increased calorific value, which can enhance the burning rate of the briquettes. Another factor to consider in briquette production is the density of the briquettes. It significantly affects their durability under load, impacting the storage and packaging process during commercialization. Table 1 presents the quality standards for briquettes across various countries.

The quality of bio-briquettes is significantly affected by various factors: (a) the type of raw materials, (b) particle size, (c) type of adhesive and its concentration, (d) pressing pressure, and (e) additional treatments, such as immersion in used cooking oil. Tapioca adhesive is commonly utilized due to its strong adhesive properties. However, tapioca, which has a low calorific value, will reduce the total calorific value of the briquette. Furthermore, the use of tapioca in large quantities will affect food reserves. Therefore, alternative adhesives are explored. Resin gum contains a lengthy carbon chain, suggesting that its utilization as an adhesive may enhance the calorific value of bio-briquettes. Haryanti, *et al.* (2023), found that utilizing resin gum as a briquette adhesive enhanced the calorific value, ranging from 7,645 to 8,731 cal/g. In addition, soaking briquettes in used cooking oil can enhance its calorific value, as it possesses a relatively high calorific value of 9,197.29 cal/g (Ariyanti & Mirwan, 2022).

This study converted tea fluffs into biochar and subsequently formed them into bio-briquettes. The response

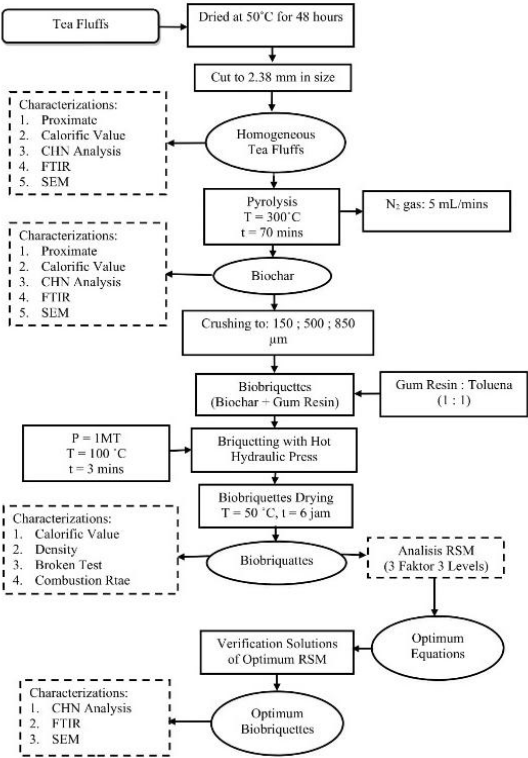


Fig. 1 Schematic diagram of the research

surface (RSM) methodology was employed to optimize the variables of the bio-briquette. In general, there were four sequential steps, starting with constructing a statistically designed experiment, forming and evaluating a predictive model using multiple regression analysis, predicting the optimum value using response surface analysis, and verifying and confirming the predictive model. The Box-Behnken design (BBD) served as the experimental framework for optimizing the analytical method. BBD requires fewer experimental combinations compared to the Central Composite Design (CCD) while effectively predicting optimal values (Nursal *et al.*, 2019, Hidayat, *et al.*, 2020). The observed variables for bio-briquette quality included particle size, resin gum concentration, and soaking time in used cooking oil. These variables were analyzed using ANOVA.

Therefore, the objectives of this study were to characterize tea fluffs before and after pyrolysis, evaluate the characteristics of the resulting briquettes, analyze the influence of briquette variables on briquette quality using ANOVA, identify optimum

Table 1
The Quality Standards of Briquette in Several Countries

Charcoal Briquette Properties	Quality Standard			
	Japan	English	America	SNI 01-6235-2000
Water Content (%)	6-8	3.6	6.2	≤8
Ash Content (%)	3-6	8.3	8.3	≤8
Volatile Matter (%)	15-30	16.4	19-28	≤15
Fixed Carbon (%)	60-80	75.3	60	≥77
Calorific Value (cal/g)	6,000-7,000	7,289	6.230	≥5,000
Burning Rate (g/min)	-	-	-	-
Density (g/cm) ³	1-1.2	0.48	1	0,447
Compressive Strength (N/cm)	60-65	12.7	62	-

Source: Research Data (2024)

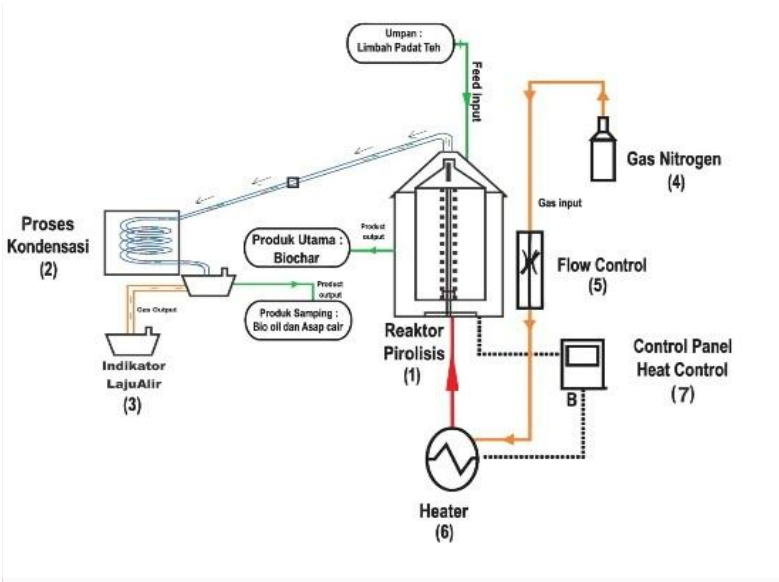


Fig. 2 Schematic of 2.5 L Capacity Pyrolysis Unit
(Source: Personal Documentation)

- Description:
1. Pyrolysis reactor

2. Ice bath/condenser

3. N gas detector2

4. N Gas Cylinder2

5. Indicator

6. N gas heater2

7. Control panel

Table 2
Research Variables Used in the Optimization of Tea Fluff Biobriquettes Manufacturing

Level	Code	Research Variables		
		Particle Size (μm)	Adhesive Concentration (%)	Dyeing Time (minutes)
Low	-1	150	10	0
Center	0	500	15	3
High	+1	850	20	6

Source: Research Data (2024)

conditions based on the RSM approach using BBD and determine the factors that have the most significant influence on the characteristics of the bio-briquettes.

2. Materials and methods

Sample preparation and characterizations are presented in Figure 1.

2.1 Tea Fluffs Preparation

The tea fluffs were gathered from the Tea and Cinchona Research Centre in Bandung, West Java. Initially, the tea fluffs were separated from impurities, including dry leaves and small stones, and subsequently dried for two days at 50°C. The tea fluffs were subsequently crushed and sieved to a size of 2.38 mm. The characterization of this raw material involved proximate analysis, calorific value assessment, CHN analysis, FTIR, and SEM.

2.2 Tea Fluff Biochar

Tea fluff biochar was produced using a pyrolysis unit with a capacity of 2.5 L (Figure 2) at 300°C and a holding time of 70 minutes (Sulaswatty, et al. 2023) (Yudo & Husman, 2019). The

biochar was characterized using proximate parameters, calorific value, CHN analysis, FTIR, and SEM.

2.3 Tea Fluff Bio-briquettes

This approach utilized the Box-Behnken Design (BBD) model, incorporating three bio-briquettes parameters: particle size, adhesive concentration, and soaking time, as detailed in Table 2, alongside the research design outlined in Table 3. In briquetting, biochar was ground into three particle sizes: 150 μm , 500 μm , and 850 μm . Subsequently, biochar was combined with resin adhesive in varying proportions of 10%, 15%, and 20%. The bio-briquettes were molded with a hot hydraulic press machine, applying a pressure of 1 metric ton at 100°C for 3 minutes. After that, the bio-briquettes were dried in an oven for 12 hours at 50°C. The dried bio-briquettes were subsequently immersed in used cooking oil for three distinct durations: 0, 3, and 6 minutes. Afterward, the briquettes were dried in an oven at 50°C for 6 hours. Finally, the bio-briquettes were analyzed for proximate analysis, calorific value, density, burning rate, and compressive strength.

2.4 ANOVA Analysis and Evaluation Predictive Model

The briquette characterization results were analyzed through Statistical Analysis of Variance (ANOVA) to determine the effect

Table 3
Research Design of Tea *Fluff* Biobriquettes Optimization

Standard Order	Particle Size (μm)	Adhesive Concentration (%)	Dyeing Time (minutes)
1	150	10	3
2	850	10	3
3	150	20	3
4	850	20	3
5	150	15	0
6	850	15	0
7	150	15	6
8	850	15	6
9	500	10	0
10	500	20	0
11	500	10	6
12	500	20	6
13	500	15	3
14	500	15	3
15	500	15	3

Source: Research Data (2024)

of variables' significance on bio-briquettes' quality. Each variable will be analyzed for each response: moisture content, ash content, volatile matter, fixed carbon, calorific value, density, burning rate, and compressive strength of the bio-briquettes. The analysis results show an optimal condition for producing tea fluff bio-briquettes by selecting solutions based on desirability values and prioritizing response values.

2.5 Verification Stage

The verification stage of bio-briquettes under optimal conditions was conducted three times. Subsequently, the data verification results were compared with the predicted values provided by the RSM solution. The analysis comprised proximate analysis, calorific value, combustion rate, density, compressive strength, CHN analysis, FTIR analysis, and SEM analysis.

3. Results and discussion

3.1 Proximate Analysis of Tea Fluffs and Its Biochar

Table 4 presents the proximate analysis and calorific value of tea fluffs before and after pyrolysis. The moisture content and volatile matter of tea fluff decreased after pyrolysis, from 7.02% to 3.05% and from 55.45% to 19.56%, respectively. In contrast, the ash content increased from 5.72% to 11.48%. The pyrolysis process, influenced by temperature and holding time, can lead to this occurrence. Increased temperature and extended pyrolysis holding time prolong combustion, leading to water evaporation and reduced moisture content. Nonetheless, the ash produced may be greater (Redjeki et al., 2023). Iskandar & Rofiatin (2017) stated that the percentage of moisture content, ash content, and volatile matter is inversely proportional to the fixed carbon and calorific value. A lower moisture content, ash content, and volatile matter correspond to higher fixed carbon and calorific values in briquettes. It can be seen in Table 4, after

pyrolysis, the fixed carbon obtained increased from 31.81% to 65.90%, as well as the calorific value, which increased from 4,482.56 cal/g to 6,374.98 cal/g. Research by Nisavira et al., (2023), yielded similar findings, indicating that the calorific value of cardamom husk and cardamom seed residue increased due to pyrolysis conducted at temperatures ranging from 250 to 450°C. This study indicates that the rise in fixed carbon and calorific value may result from the carbonization process employed through the pyrolysis method. Therefore, the pyrolysis process influences the proximate and calorific values of biochar.

3.2 Bio-briquetting Optimization Analysis

The optimization results of tea fluff bio-briquettes are detailed in Table 5. Table 5 shows that the moisture content of tea fluff bio-briquettes ranges from 3.11-6.45%, ash content 5.76-10.91%, volatile matter 15-19.49%, fixed carbon 63.51-76.14%, calorific value 6,502.03-7,749.66 cal/g, burning rate 0.06-0.14 g/min, density 0.59-1.22 g/cm³, and compressive strength 12.31-106.34 N/cm². The characteristics of tea fluff bio-briquettes comply with SNI 01-635-2000 regarding moisture content, ash content, and calorific value, and also meet Japanese quality standards for volatile matter and fixed carbon.

The characteristics of tea fluff bio-briquettes were analyzed using ANOVA. This analysis involved assessing the significance of the model, lack of fit, and fit statistics, including R² and adjusted R². The findings are depicted in Table 6. ANOVA analysis was employed to assess the significance of all variables in the response of the developed model. The significance of a model was assessed by analyzing the F-value generated and p-value. Table 6 indicates that the F-value of the resulting model exceed 0.05, while the p-value was less than 0.05. Tasfiyati et al., (2022) reported that a model is considered statistically significant if the F-value exceeds 0.05 (F>0.05) and the p-value is less than 0.0001. However, in a similar study Sutarna et al. (2023) indicated that a model is considered significant when the

Table 4
Proximate Analysis and Calorific Value of Tea Fluffs Before and After Pyrolysis

Treatment	Sample	Water Content (%)	Ash Content (%)	Volatile Matter (%)	Fixed Carbon (%)	Calorific Value (%)
Before pyrolysis	Tea Fluffs	7.02	5.72	55.45	31.81	4,482.56
After pyrolysis (Biochar)		3.05	11.48	19.56	65.90	6,374.98

Source: Research Data (2024)

Table 5
Characterization Results of Tea *Fluff* Bio-briquettes

Std	Particle Size (µm)	Adhesive Concentration (%)	Dyeing Time (minutes)	Water Content (%)	Ash Content (%)	Volatile Matter (%)	Fixed Carbon (%)	Calorific Value (cal/g)	Burning Rate (g/min)	Compressive strength (N/cm ²)	Density (g/cm ³)
1	150	10	3	3.45	6.32	15.53	74.71	7671.29	0.14	12.31	1.02
2	850	10	3	3.11	5.76	15.00	76.14	7732.17	0.12	27.83	1.10
3	150	20	3	5.64	7.29	16.42	70.65	7664.92	0.10	40.98	1.15
4	850	20	3	4.96	6.70	16.39	71.96	7553.44	0.11	61.90	1.01
5	150	15	0	6.10	10.91	19.49	63.51	6587.61	0.13	95.13	0.65
6	850	15	0	5.80	9.69	19.03	65.48	6584.93	0.10	94.52	0.60
7	150	15	6	4.10	6.45	15.74	73.71	7749.66	0.12	28.19	1.06
8	850	15	6	4.18	6.70	15.35	73.78	7656.37	0.11	52.28	1.09
9	500	10	0	4.99	10.72	18.75	65.54	6502.03	0.13	44.46	0.59
10	500	20	0	6.45	10.47	19.21	63.87	6723.17	0.06	106.34	0.63
11	500	10	6	3.85	6.03	15.28	74.85	7662.03	0.12	13.46	1.22
12	500	20	6	5.32	6.83	15.89	71.97	7613.17	0.12	22.45	0.99
13	500	15	3	4.50	6.32	15.45	73.73	7589.93	0.11	18.79	1.05
14	500	15	3	4.40	6.24	15.34	74.01	7607.63	0.11	18.78	1.10
15	500	15	3	4.63	6.56	15.79	73.03	7524.58	0.10	26.63	1.00

Source: Research Data (2024)

resulting p-value is <0.05. Consequently, prior research findings support the assertion that the model established in this study demonstrates significance. The p-value <0.05 in the model indicates that the dependent variable affects the independent variable.

The appropriateness of the regression model is further evaluated through the lack of fit test. Table 6 indicates that the

lack of fit test is inversely related to the regression model, with the resulting lack of fit test demonstrating insignificance, as evidenced by a p-value >0.05. The lack of fit test evaluates the difference between predicted values and actual data. The most desirable condition is a lack of significant fit, indicating that the developed model is effective and aligns well with the response data. This aligns with the research by Tasfiyati *et al.*, (2022),

Table 6
ANOVA (Analysis of Variance) Results of Tea *Fluff* Bio-briquettes

Response	Model			Lack of fit			Fit statistic	
	F-value	p-value	Description	F-value	p-value	Description	R ²	Adjusted R ²
Water Content	21.15	0.0003	Significant	8.54	0.1081	not significant	0.9548	0.9097
Ash Content	111.61	< 0.0001	Significant	2.73	0.2893	not significant	0.9911	0.9822
Volatile Matter	57.95	0.0002	Significant	1.56	0.4137	not significant	0.9905	0.9734
Fixed Carbon	58.64	0.0002	Significant	2.47	0.3010	not significant	0.9906	0.9737
Calorific Value	88.08	< 0.0001	Significant	3.40	0.2426	not significant	0.9888	0.9775
Density	32.84	0.0076	Significant	0.0554	0.8358	not significant	0.9918	0.9616
Burning Rate	28.64	0.0009	Significant	1.21	0.4823	not significant	0.9810	0.9467
Compressive Strength	103.53	< 0.0001	Significant	0.5408	0.7003	not significant	0.9947	0.9851

Source: Research Data (2024)

Table 6
Quadratic Regression Equation

Response	Estimation of Selected Coefficients in the Form of Quadratic Regression Equation
Water Content	Y = 4.38 - 0.1550 A + 0.8712 B - 0.7363 C - 0.0850 AB + 0.0950 AC + 0.0025 BC + 0.7145 C ²
Ash Content	Y = 6.46 - 0.2650 A + 0.3075 B - 1.97 C - 0.0075 AB + 0.3675 AC + 0.2625 BC + 2.02 C ²
Volatile matter	Y = 15.53 - 0.1771 A + 0.4186 B - 1.78 C + 0.1233 AB + 0.0134 AC + 0.0368 BC + 0.2125 A ² + 0.0924 B ² + 1.66 C ²
Fixed carbon	Y = 73.59 + 0.5967 A - 1.60 B + 4.49 C - 0.0292 AB - 0.4754 AC - 0.3029 BC - 0.0822 A ² - 0.1454 B ² - 4.39 C ²
Calorific Value	Y = 7620.56 - 18.32 A - 1.60 B + 535.44 C - 43.09 AB - 22.65 AC - 67.50 BC - 485.69 C ²
Burning Rate	Y = 0.1070 - 0.0107 A - 0.0188 B + 0.0117 C + 0.0085 AB + 0.0059 AC + 0.0158 BC + 0.0073 A ² + 0.0026 B ² + 0.0076 A ² B - 0.0103 A ² C + 0.0069 AB ²
Density	Y = 1.05 - 0.0090 A - 0.0205 B + 0.2360 C - 0.0551 AB + 0.0182 AC - 0.0658 BC + 0.0054 A ² + 0.0144 B ² - 0.2051 C ²
Compressive Strength	Y = 21.40 + 7.49 A + 16.70 B - 28.01 C + 1.35 AB + 6.17 AC - 13.22 BC + 17.60 A ² - 3.254 B ² + 28.53 C ²

Source: Research Data (2024)

Description: A = Biochar particle size , B = Resin Gum Adhesive Concentration , C = Dipping Time in Used Cooking Oil

which indicates that a lack of fit with a p-value >0.05 and non-significance suggests that the prediction model is reasonably appropriate for the experimental data.

The adequacy of a model can also be seen from the R-squared (R^2) and adjusted R^2 (Jegan *et al.*, 2021). The R^2 and adjusted R^2 values for each response in this study were notably high, exceeding 0.90 (Table 6). High R^2 and adjusted R^2 values signify a strong correspondence between predictions and actual experimental data. R^2 is considered high if it falls within the range of 0 to 1. A higher R^2 value signifies a more effective model (Baharin *et al.*, 2023, Irmawati *et al.*, 2023).

The quadratic regression equation selected for each response is presented in Table 7. Table 7 indicates that the immersion time of tea fluff bio-briquettes in used cooking oil significantly influences ash content, volatile matter, fixed carbon, calorific value, density, and compressive strength. The resin gum adhesive concentration factor significantly influences the moisture content and burning rate of tea fluff bio-briquettes. A higher estimated coefficient between variables indicates a stronger tendency of the factor's influence. For instance, in the moisture content response, the adhesive concentration factor (variable B) exhibits a higher value compared to the particle size

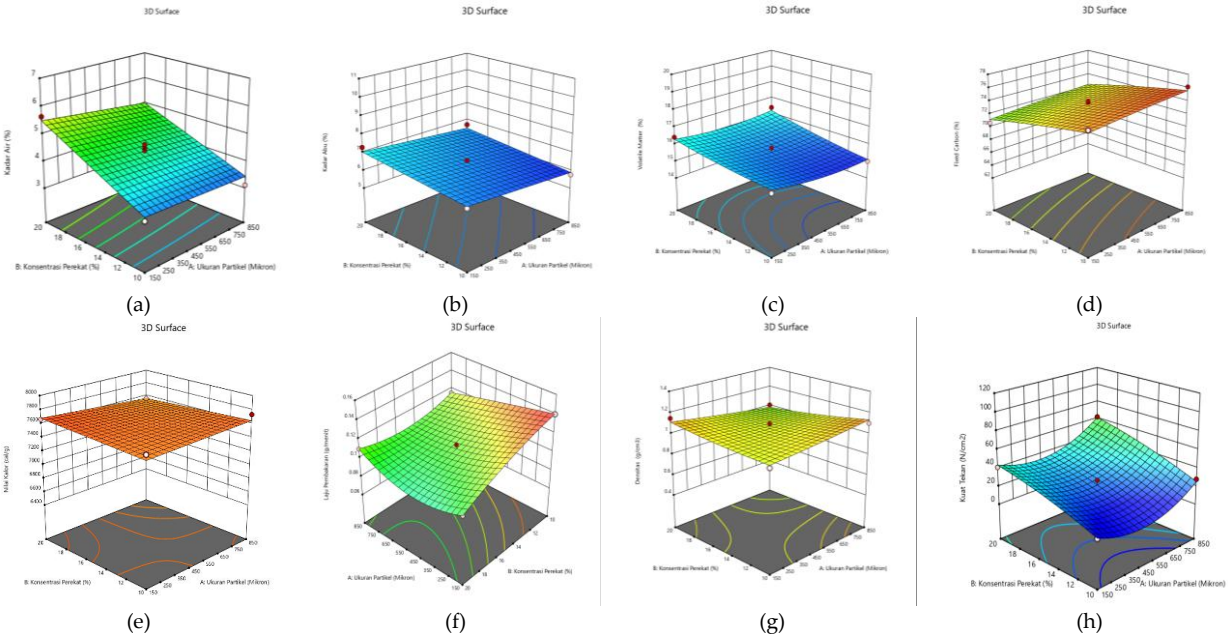


Fig. 3 Interaction between Biochar Particle Size and Adhesive Concentration Factors on the Response of (a) Moisture Content, (b) Ash Content, (c) Volatile Matter, (d) Fixed Carbon, (e) Calorific Value, (f) Burning Rate, (g) Density, and (h) Compressive Strength of Tea Fluff Biobriquettes in 3D Surface.

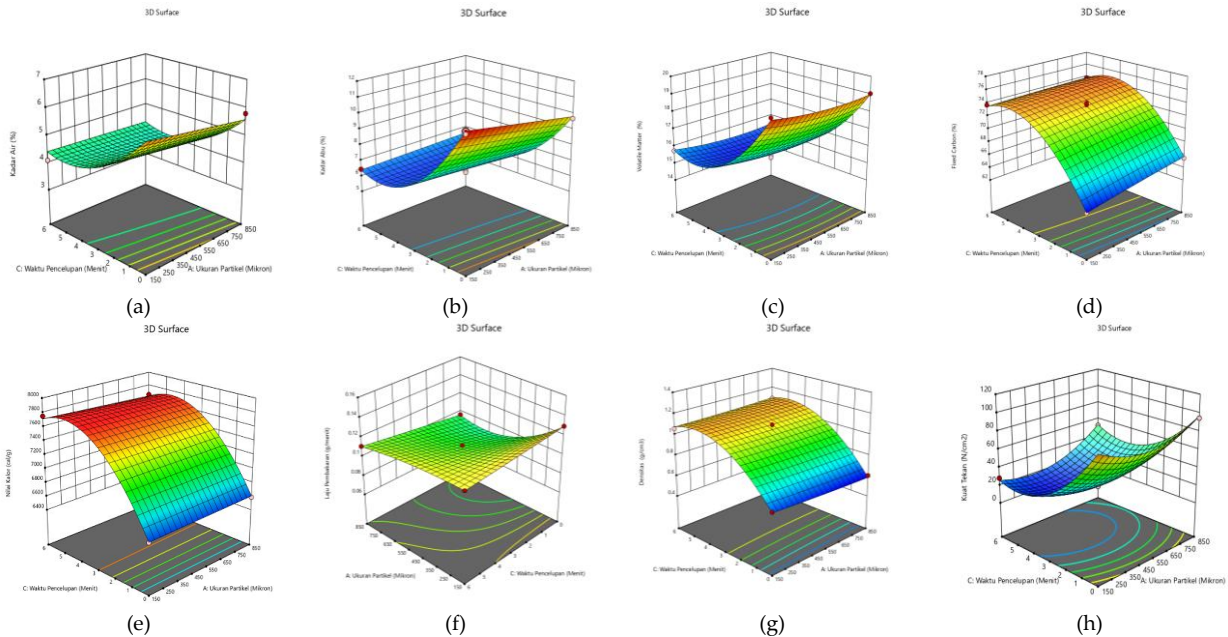


Fig. 4 Interaction between Factors of Biochar Particle Size and Immersion Time in Used Cooking Oil on the Response of (a) Moisture Content, (b) Ash Content, (c) Volatile matter, (d) Fixed Carbon, (e) Calorific Value, (f) Burning Rate, (g) Density, and (h) Compressive Strength of Tea Fluff Biobriquettes in 3D Surface Form.

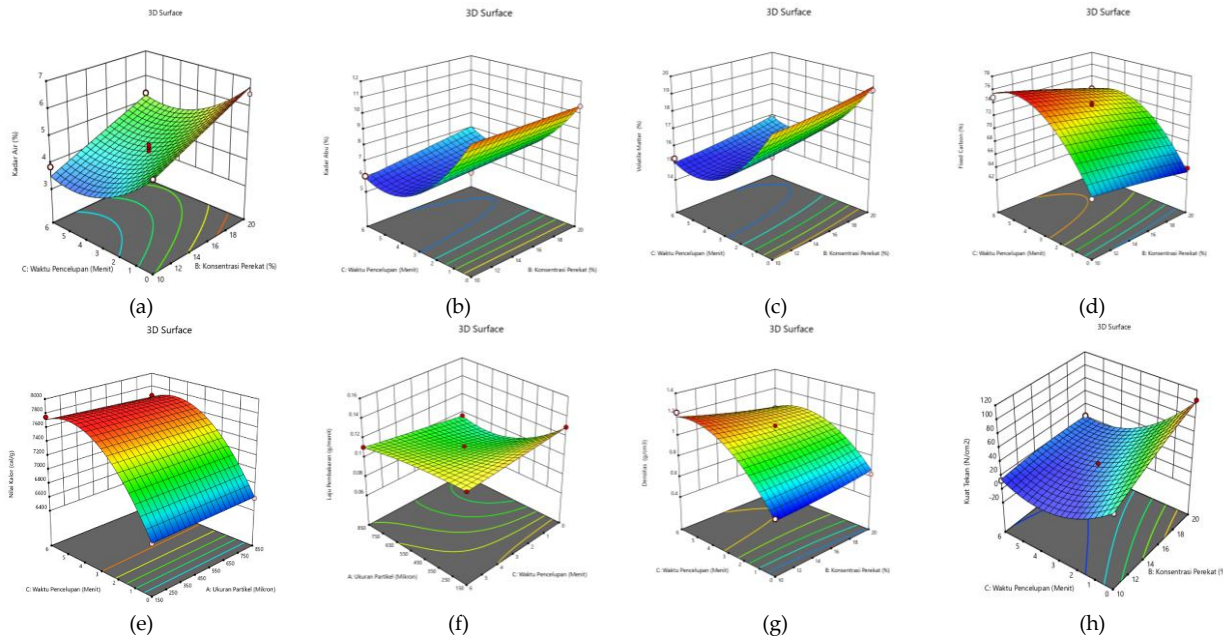


Fig. 5 Interaction between the Factors of Adhesive Concentration and Immersion Time in Used Cooking Oil on the Response of (a) Moisture Content, (b) Ash Content, (c) Volatile Matter, (d) Fixed Carbon, (e) Calorific Value, (f) Burning Rate, (g) Density, and (h) Compressive Strength of Tea Fluff Biobriquettes in 3D Surface Shape

factor (variable A) and the immersion time in cooking oil (variable C). The positive and negative signs associated with each variable denote whether the influence of factors is directly or inversely proportional. Limahelu *et al.* (2021) found that the positive sign on the variable value suggests a direct proportionality between the influence and the response. This implies an increased variable coefficient leads to a more significant/higher resulting response value.

The negative sign on the variable value indicates that the influence tends to be inversely proportional to the response. The greater the variable coefficient employed, the lower the

resulting response value. Figure 3-5 illustrates the interaction among biochar particle size, resin gum adhesive concentration, and immersion time of tea fluff bio-briquettes, focusing on their effects on moisture content, ash content, volatile matter, fixed carbon, calorific value, combustion rate, density, and compressive strength.

3.2 The Variable of Biochar Particle Size

Figure 6 is a one-factor graph that shows the trend of the effect of biochar particle size factor on proximate value, calorific value,

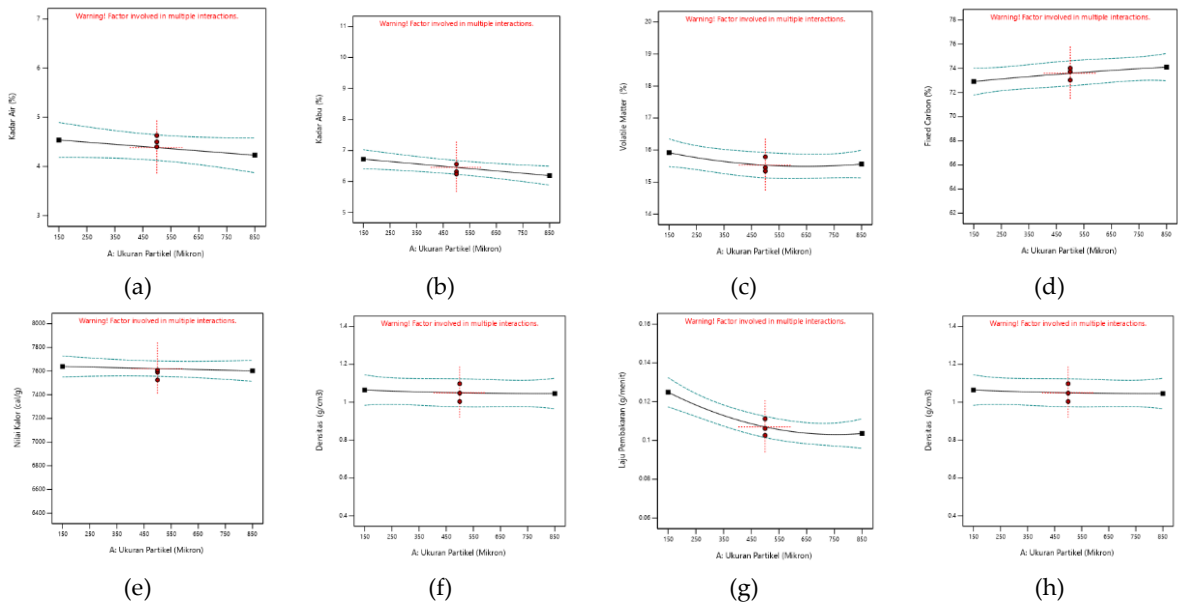


Fig. 6 Trend Graph of the Effect of Particle Size Factor for the Response of (a) Moisture Content, (b) Ash Content, (c) Volatile Matter, (d) Fixed Carbon, (e) Calorific Value, (f) Density, (g) Burning Rate, (h) Compressive Strength of Tea Fluff Biobriquettes.

combustion rate, density, and compressive strength of tea fluff briquettes. Figures 6a-6e and the signs of the estimated coefficients for variable A in the regression equation in Table 7 indicate that particle size inversely affects moisture content, ash content, volatile matter, and heating value. However, it has a direct proportional impact on the fixed carbon content of the briquettes. Larger particle sizes correspond to lower moisture content, ash content, volatile matter, and heating value, while fixed carbon content increases. Smaller particle sizes in briquettes result in smaller pores, which hinder the evaporation of water during the drying process. Consequently, the moisture content is greater than that of bio-briquettes with larger particle sizes (Alfajriandi et al., 2017). Iskandar, et al (2019) found that a decrease in the moisture content of bio-briquettes corresponds to a reduction in the volatile matter.

Furthermore, during combustion, bio-briquettes with smaller particle sizes exhibit more complete burning, resulting in greater ash production than those with larger particle sizes (Situmorang & Kusmartono, 2022). Putri & Andasuryani (2017) indicated that moisture content, ash content, and volatile matter significantly influence the fixed carbon content and heating value of bio-briquettes. A lower moisture content, ash content, and volatile matter correspond to a higher fixed carbon content, resulting in an increased heating value (Putri & Andasuryani, 2017). However, this study found that the heating value value is inversely proportional to the fixed carbon content of the briquettes. Larger biochar particle sizes correlate with increased fixed carbon content. However, the heating value produced is lower. This can occur because bio-briquettes with smaller particle sizes have a larger energy transfer area, resulting in a higher heating value than bio-briquettes with larger particle sizes (Ristianingsih et al., 2013).

The quality of bio-briquettes is assessed based on their proximate value, calorific value, density, burning rate, and compressive strength. Figure 6f and the negative coefficient estimate for variable A in the regression equation in Table 7 indicate that particle size inversely affects the density of the briquettes. Larger particle sizes result in lower briquette density, while smaller particle sizes lead to higher density. Briquettes of smaller particle sizes exhibit reduced pore sizes, resulting in a

tighter and more compact bond between particles. Qanitah et al., (2023) indicated that briquette density influences both the burning rate and compressive strength of the briquettes. The high density of briquettes enhances their resistance to pressure. However, igniting is generally more challenging due to the denser and more compact bonds between particles, which restrict air entry and result in a slower burning rate (Haryanti et al., 2023).

However, in this study, the results obtained were contrary. The particle size exhibits an inverse relationship with the burning rate of briquettes, as illustrated in Figure 6g. It demonstrates a direct relationship with the compressive strength of briquettes, as shown in Figure 6h. This means that larger particle sizes result in slower burning rates and increased compressive strength of the briquettes. The addition of adhesive concentration may lead to this discrepancy. Furthermore, the pressure exerted during the compressive strength test with the Universal Testing Machine (UTM) may influence compressive strength. Larger particle sizes undergo re-compaction, resulting in denser particle arrangements and higher compressive strength values for the briquette (Setyopambudi, 2015). Ariyanti & Mirwan, (2022) discovered that an increased pressing level enhances the binding of the adhesive and charcoal components, resulting in a stronger briquette.

3.3 The Variable of Resin Gum Concentration

Figure 7 is a one-factor graph that illustrates the trend of the effect of resin gum adhesive concentration on the proximate value, calorific value, burning rate, compressive strength, and density of tea fluff biobriquettes. Figures 9a-9c and the positive coefficient estimate for variable B in the regression equation in Table 7 indicate that the concentration of resin gum adhesive is directly proportional to moisture content, ash content, and volatile matter. This suggests that an increase in the concentration of resin gum adhesive correlates with higher levels of moisture content, ash content, and volatile matter. The findings are consistent with the research by Zuhry et al. (2022), which indicated that the moisture content, ash content, and volatile matter in maltodextrin-adhesive dry leaf waste

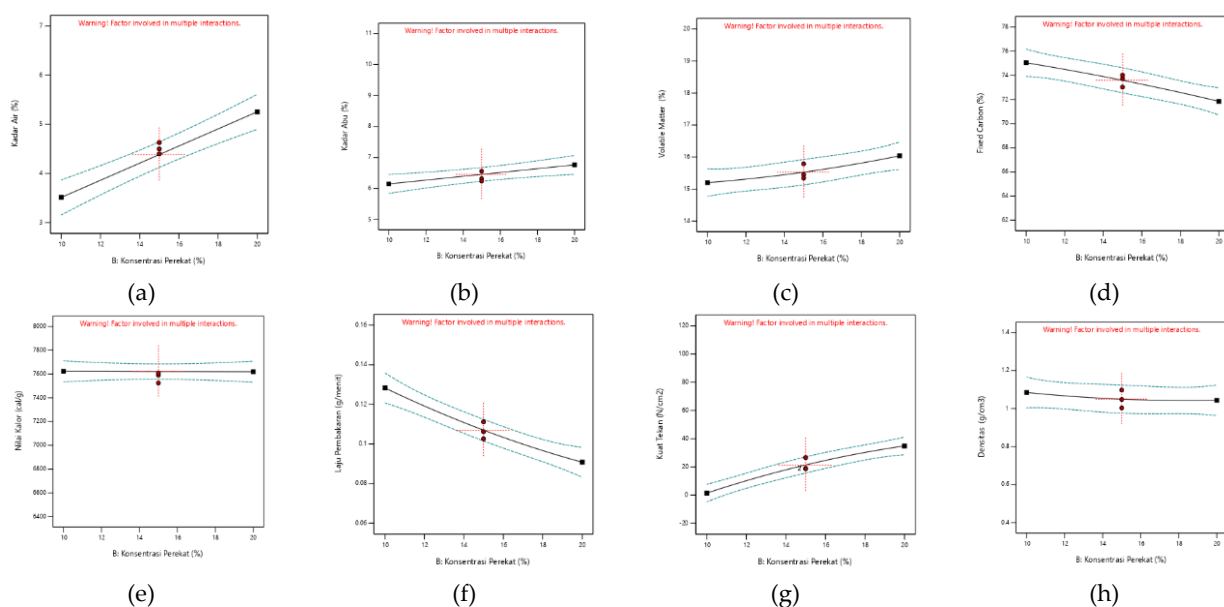


Fig. 7 Trend Graph of the Effect of Adhesive Concentration Factor for the Response of (a) Moisture Content, (b) Ash Content, (c) Volatile Matter, (d) Fixed Carbon, (e) Calorific Value, (f) Burning Rate, (g) Compressive Strength, (h) Density of Tea Fluff Biobriquettes.

briquettes increased with higher concentrations of adhesive. The increase might be caused by the additional water and ash content from the adhesive. Furthermore, the volatile components in the adhesive, including hydrogen, carbon dioxide, and methane, evaporate during combustion, thereby increasing the volatile matter in the briquettes.

The concentration of resin gum adhesive negatively impacts the fixed carbon content and heating value of the briquettes, as evidenced by the negative coefficient for variable B in the regression equation shown in Table 7, along with Figures 9d and 9e. In other words, as the adhesive concentration increases, the fixed carbon content and heating value decreases. The phenomenon arises from the increasing moisture content, ash content, and volatile matter, which decrease the fixed carbon content, resulting in a lower calorific value of the bio-briquettes.

The concentration of adhesives influences the burning rate, density, and compressive strength of the briquettes. Figure 7f and the negative coefficient of variable B in the regression equation in Table 7 indicate that adhesive concentration influences the burning rate response. An increase in adhesive concentration results in a decrease in the burning rate. The increased concentration of adhesives in bio-briquette results in a stronger bond between the particles and the adhesive, thereby impeding air entry. As a result, the bio-briquettes will exhibit increased difficulty in combustion. The low calorific values also influence the burning rate of the bio-briquettes. Masthura *et al.* (2022) indicated that higher heating values might accelerate the combustion rate and raise the enthalpy value during combustion. Applying a substantial quantity of adhesive can positively influence the compressive strength of bio-briquettes. Figure 7g and the positive coefficient estimate for variable B in the regression equation in Table 7 indicate that the adhesive concentration directly influences the compressive strength of tea fluff bio-briquettes. Increased adhesive concentration correlates with enhanced compressive strength of tea fluff bio-briquettes, which can beneficially affect the packaging and shipping of these products upon commercialization.

This study identified varying density effects through the addition of adhesive concentration. Figure 7h and the negative coefficient of variable B in Table 7 indicate that adhesive

concentration has an inversely proportional effect on the density response of the bio-briquettes. The more adhesive concentration used, the lower the density of the briquettes. The density and moisture content of briquettes are interrelated factors that can contribute to this issue. Yuliza *et al.* (2013) found that the density of briquettes is closely associated with their moisture content; specifically, lower moisture content results in higher briquette density. In contrast, an increase in moisture content results in a decrease in the density of the briquettes. The moisture content, whether high or low, is affected by the concentration of the adhesive applied. This study found that moisture content rises as adhesive concentration increases. Consequently, adjusting the adhesive concentration to achieve briquettes of the desired quality is essential.

3.4 The Variable of Immersion Time in Used Cooking Oil

Figure 8 is a one-factor graph that shows the trend of the influence of the immersion time factor in cooking oil on the proximate value, calorific value, combustion rate, density, and compressive strength of tea fluffs biobriquettes. Based on Figures 10a-10c and the negative coefficient of variable C in the regression equation from Table 7 indicate that the immersion time of tea fluff bio-briquettes in used cooking oil inversely affects moisture content, ash content, and volatile matter. Longer immersion times of tea fluff bio-briquettes in used cooking oil result in decreased moisture content, ash content, and volatile matter. The immersion of bio-briquettes in used cooking oil results in oil absorption by the pores of the bio-briquettes, leading to an oil coating on their surface. Consequently, the briquettes exhibit increased difficulty in absorbing the surrounding water. As a result, the moisture content of bio-briquettes immersed in used cooking oil is lower than those without immersion (Efelina *et al.*, 2018). This condition also influences the lower ash and volatile matter content. This study demonstrates that cooking oil immersion enhances the quality of tea fluff bio-briquettes by resulting in lower moisture content, ash content, and volatile matter compared to the absence of cooking oil immersion.

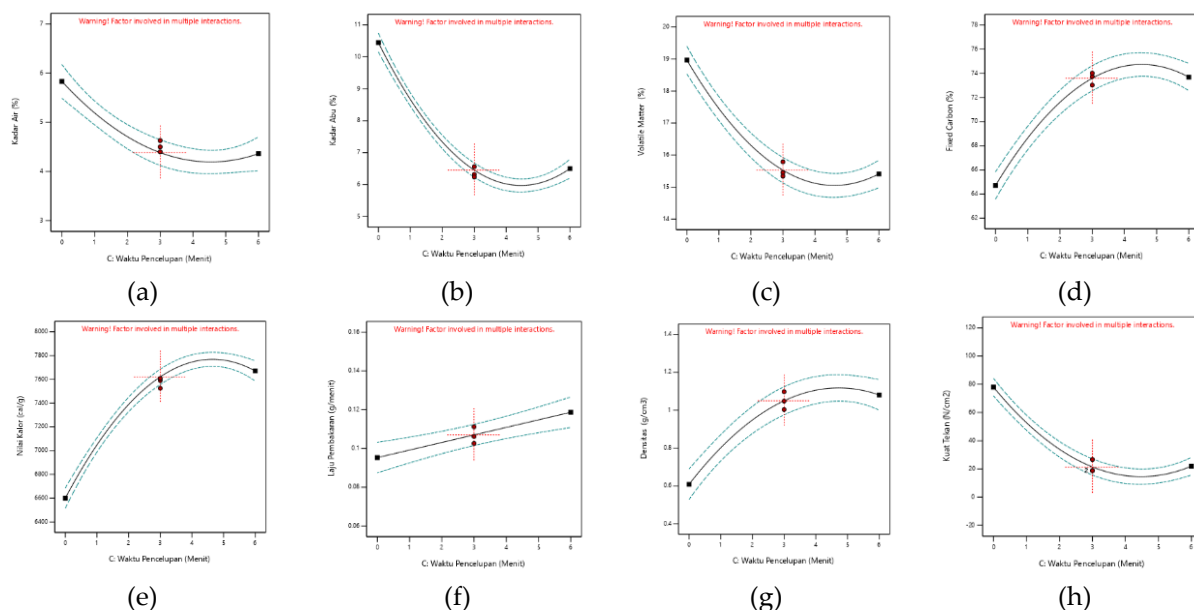


Fig. 8 Trend Graph of the Effect of Dipping Time Factor for the Response of (a) Moisture Content, (b) Ash Content, (c) Volatile Matter, (d) Fixed Carbon, (e) Calorific Value, (f) Burning Rate, (g) Density, (h) Compressive Strength of Tea Fluff Biobriquettes.

Table 7
Solution and Predicted Value of RSM

Sample	Particle Size (μm)	Adhesive Concentration (%)	Dyeing Time (minutes)	Response	Predicted Value Design Expert
Tea Fluff Biobriquettes	850	11	5	Water Content (%)	5.53
				Ash Content (%)	5.65
				Volatile Matter (%)	14.75
				Fixed Carbon (%)	76.14
				Calorific Value (cal/g)	7,796.37
				Burning Rate (g/min)	0.11
				Density (g/cm ³)	1.22
				Compressive Strength (N/cm ²)	35.57

Source: Research Data (2024)

The quality of bio-briquettes is primarily determined by their fixed carbon content. Figure 8d and the positive coefficient of variable C in the regression equation from Table 7 indicate that the immersion time of tea fluff bio-briquettes in used cooking oil directly affects the response of fixed carbon. Increased immersion time of bio-briquettes in used cooking oil results in higher fixed carbon content. This occurs due to reduced moisture, ash, and volatile matter when immersed in used cooking oil. Putri & Andasuryani, (2017) found that the level of fixed carbon is significantly influenced by moisture content, ash content, and volatile matter, exhibiting inversely proportional relationships. The relationship indicates that a decrease in moisture content, ash content, and volatile matter corresponds to an increase in fixed carbon and vice versa. The immersion time of bio-briquettes in used cooking oil also directly affects the calorific value, combustion rate, and density of tea fluff bio-briquettes. Figure 8e to Figure 8g, along with the positive coefficient value of variable C in the regression equation shown in Table 7, indicates that the immersion time of tea fluff bio-briquettes in used cooking oil tends to influence the heating value, burning rate, and density of the bio-briquettes in a directly proportional manner. This means that the longer the immersion time of the bio-briquettes in used cooking oil, the higher the heating value, combustion rate, and density of the bio-briquettes. The immersion of briquettes in used cooking oil results in the absorption of the oil by the pores.

Consequently, the briquettes exhibit greater difficulty absorbing surrounding water, leading to a lower moisture content than those not subjected to cooking oil immersion. The combination of low moisture content and high carbon content enhances the calorific value of bio-briquettes and accelerates their combustion rate. Furthermore, the burning rate of the briquettes is more regulated due to their oil coating, which reduces their hygroscopic properties (Efelina et al., 2018). In addition, briquettes with low moisture content also have a high density (Yuliza et al., 2013).The cooking oil used as an immersion medium for bio-briquettes is an additional adhesive, enhancing the bond between bio-briquette particles. This results in tighter and more compact pores, thereby increasing the compressive strength of the bio-briquettes.

In this study, the compressive strength value was inversely proportional to the density of the briquettes, as evidenced in Figure 8h, and the negative sign on the estimated coefficient of Variable C. The longer the immersion time of bio-briquettes in used cooking oil, the lower the compressive strength of bio-briquettes. Nevertheless, the briquettes maintained their shape and did not become crushed or split in half. The study's findings are consistent with the research by Ariyanti & Mirwan, (2022), which indicated that the optimal compressive strength was achieved in briquettes immersed for 5 and 10 minutes, as opposed to those immersed for 20 minutes. The immersion time

of bio-briquettes in used cooking oil significantly influences their integrity and solidity, necessitating an optimal immersion duration.

3.5 Solution of the RSM approach

Table 8 indicates that the optimal conditions for producing tea fluff bio-briquettes, as determined by RSM, are a biochar particle size of 850 μm , a resin gum adhesive concentration of 11%, and an immersion time in cooking oil of 5 minutes. The selected condition is based on a desirability value of 0.787 (78.7%), as it yields predicted values for moisture content, ash content, volatile matter, and heating value that comply with the Indonesian National Standard (SNI) 01-6235-2000. Additionally, it ensures that the fixed carbon, density, and compressive strength of briquettes meet the quality standards of Japan, England, and the United States, with a briquette combustion rate of 0.11 g/minute. The quality standards for briquettes, including SNI 01-6235, as well as Japanese, British, and American standards, are presented in Table 1. This study also determines the optimal conditions for producing tea fluff bio-briquettes based on the highest calorific value. The higher the calorific value, the better the quality of the bio-briquettes.

3.6 Biobriquette Verification Results

Data verification aimed to assess whether the response value from the verification results aligns with the predicted value provided by the RSM solution. The verification stage was conducted three times, utilizing a biochar particle size of 850 μm , a resin gum adhesive concentration of 11%, and an immersion time in cooking oil of 5 minutes. Table 9 presents the average results obtained from the verification stage.

The accuracy of the average value of the verification results with the predicted value given by the RSM can be determined by calculating the accuracy level. The accuracy level is deemed acceptable if it falls within 85-115% (Tasfiyati et al., 2022). Table 9 indicates that the average value obtained from the verification stage, compared to the predicted value of the RSM solution, demonstrates an accuracy level ranging from 85% to 115% for all responses except the compressive strength response. Therefore, the verification results were considered acceptable. Regarding compressive strength response, the achieved accuracy level exceeded 115%, specifically 128.26%. Therefore, the verification results were less accurate than the predicted values generated by the RSM solution. The average value of the verification results for the compressive strength response was notably high. The average value of the verification stage results for each response met the quality standards for briquette quality, including SNI 01-6235-2000, as well as Japanese, British, and American standards.

Table 8
Average Verification Results from RSM

Sample	Particle size (μm)	Adhesive Concentration (%)	Dyeing Time (minutes)	Response	Average Verification	Solution RSM	Accuracy Level
Tea Fluff Biobriquettes	850	11	5	Water Content (%)	3.44±0.03	3.53	97.31
				Ash Content (%)	6.01±0.13	5.65	106.33
				Volatile Matter (%)	15.22±0.27	14.75	103.18
				Fixed Carbon (%)	75.34±0.39	76.14	98.95
				Calorific Value (cal/g)	7,609.03±25.68	7,796.37	97.60
				Burning Rate (g/min)	0.10	0.11	94.95
				Density (g/cm3)	1.15±0.03	1.22	94.46
				Compressive strength (g/cm2)	45.62±7.46	35.57	128.26

Source: Research Data (2024)

Table 9
CHN Analysis Results of Tea Fluffs, Tea Fluff Biochar and Tea Fluff Biobriquettes

Test Sample	CHN Analysis		
	Carbon Content (C) (%)	Hydrogen (H) Content (%)	Nitrogen (N) content (%)
Tea Fluffs	46.80	3.46	6.71
Tea Fluff Biochar	65.54	4.11	5.37
Tea Fluff Biobriquettes	67.93	2.24	7.83

Source: Research Data (2024)

3.7 Advanced Characterization Results

Additional characterization was conducted on tea fluff bio-briquettes sample produced under optimal conditions. The characteristics encompassed CHN analysis, FTIR (Fourier Transform Infrared), and SEM (Scanning Electron Microscope) analysis. CHN analysis determines the C, H, and N content of the sample. Table 10 indicates a significant C content increase, from 46.80% in tea fluffs prior to pyrolysis to 65.54% after conversion to biochar. The charring process utilizing the pyrolysis method can lead to the evaporation of OH compounds composed of hydrogen and oxygen, forming pure carbon (Bazenet *et al.*, 2021). The C content in biochar increased from 65.54% to 67.93% after being processed into briquettes. The elevated C content in biochar is a significant factor influencing bio-briquettes' quality. The higher the C content in a material, the higher the heating value is produced. Ahmad *et al.* (2023) further explained that C content influences the calorific value of fuel and hydrogen, whereas N content correlates with the level of emissions produced. The H content in tea fluffs prior to pyrolysis, following pyrolysis, and after briquetting was 3.46%, 4.11%, and 2.24%, respectively. The N content in the three samples was 6.71%, 5.37%, and 7.83%, respectively. The N content generated remained relatively low, resulting in minimal emissions.

Figure 9 presents the functional groups identified from the FTIR analysis of tea fluff samples, tea fluff biochar, and tea fluff bio-briquettes. FTIR analysis aimed to identify the functional groups present in tea fluffs before pyrolysis, in tea fluff biochar post-pyrolysis, and in tea fluff bio-briquettes under optimal conditions. FTIR analysis was performed within the wavelength range of 4000 cm⁻¹ to 400 cm⁻¹. The FTIR analysis in Figure 9 indicates that the OH functional group is exclusively found in tea fluffs prior to pyrolysis, observed at a wavelength of 3309 cm⁻¹. However, it was absent in biochar and tea fluff bio-briquettes. The charring process employed in the pyrolysis method can lead to removing OH functional groups from biochar. This disrupts bonds between O and H atoms (de

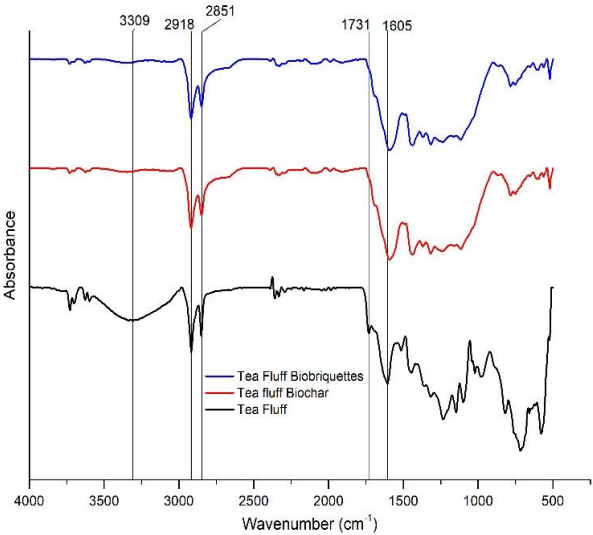


Fig. 9 Spectra FTIR Analysis Results of Tea Fluff, Tea Fluff Biochar and Tea Fluff Biobriquettes

Almeida *et al.*, 2022). This disruption leading to increased C content in biochar compared to the CH functional group in the three samples. Rahmi *et al.*, (2023) indicated that the absence of CH functional groups signified the presence of cellulose, hemicellulose, and lignin in the samples (tea fluffs, tea fluff biochar, and tea fluff bio-briquettes). The presence of the CH functional group also signifies hydrocarbon compounds in the bio-briquettes (Tiwow *et al.*, 2021). The CH functional group in tea fluff prior to pyrolysis was identified within the wavelength range of 2918-2851 cm⁻¹. In tea fluff biochar, this group was observed in the range of 2919-2849 cm⁻¹, while in the tea fluff bio-briquettes produced under optimal conditions, it was detected in the range of 2921-2852 cm⁻¹. Figure 9 indicates that the CH functional group exhibits a sharper intensity in tea fluff

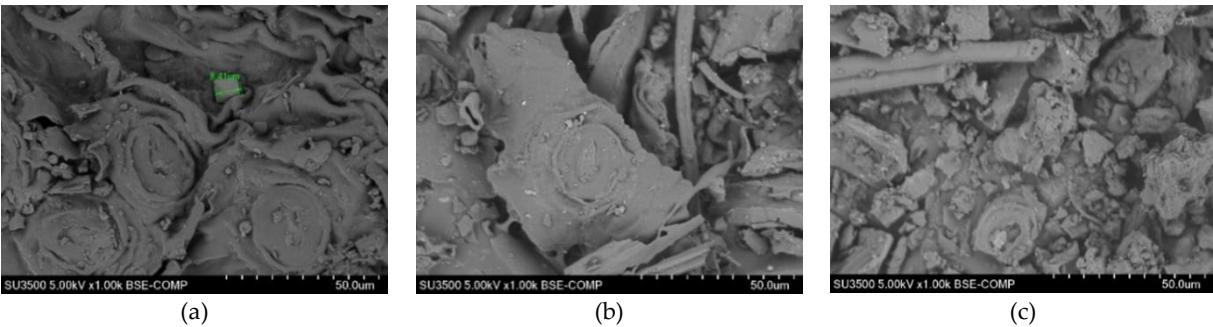


Fig. 10 SEM Analysis Results of (a) Tea Fluffs, (b) Tea Fluff Biochar, and (c) Tea Fluff Biobriquettes

briquettes compared to tea fluff biochar and the tea fluffs prior to pyrolysis. The observed phenomenon is attributed to the immersion treatment of tea fluff briquettes in cooking oil for 5 minutes, as indicated in the study by Bazenet *et al.* (2021). This treatment results in a more pronounced slope of OH and CH functional groups, which is linked to alterations in the chemical bonds of the briquettes influenced by adhesive application.

In addition, C=O bond stretching vibrations were observed in tea fluffs prior to pyrolysis at a wavelength of 1731 cm⁻¹. In tea fluff biochar, these vibrations occurred at 1691 cm⁻¹, exhibiting lower intensity compared to the tea fluffs before pyrolysis. For tea fluff briquettes produced under optimal conditions, the vibrations were detected at 1743 cm⁻¹, demonstrating sharper intensity than the tea fluffs before pyrolysis and the tea fluff biochar. The low intensity of tea fluff biochar occurs due to the pyrolysis process, which breaks the acetyl bond, a hemicellulose bond within the material (Bazenet *et al.*, 2021; W. Hidayat *et al.*, 2020). The ethanol form of C=O bond stretching vibrations was observed exclusively in tea fluffs prior to pyrolysis and in tea fluffs biochar, specifically at wavelengths of 1605 cm⁻¹ and 1592 cm⁻¹. In contrast, these vibrations were absent in tea fluff briquettes, attributed to alterations in the compound groups present in the various compositions used for their production.

This study conducted SEM-EDX (Scanning Electron Microscope-Energy Dispersive X-ray) analysis to examine the changes in structure, surface morphology, and elemental composition of tea fluff samples, tea fluff biochar, and tea fluff bio-briquettes produced under optimal conditions. SEM-EDX analysis operates by directing a high-energy electron beam at the sample, which generates an image and reveals the sample's elemental composition (Sahdiah & Kurniawan, 2023). The results are presented in Figure 10 and Table 11. The SEM analysis results (Figure 10) indicate that the samples of tea fluffs, tea fluff biochar, and tea fluff bio-briquettes exhibit distinct

surface morphologies. The surface morphology of the tea fluffs is tighter and exhibits smaller pore sizes, with C and O elements comprising 68.89% and 27.29%, respectively. Simultaneously, the tea fluff biochar exhibited an increase in porosity, marked by the enlargement of biochar pores, with C and O content at 68.17% and 26.30%, respectively.

The C content in tea fluff biochar has significantly decreased compared to that in tea fluffs. This is likely due to human error during testing. The small pore size in tea fluffs prior to pyrolysis is attributed to the presence of lignin, hemicellulose, and other components that bind cellulose. In contrast, the larger pore size in tea fluff biochar results from the charring process associated with pyrolysis, which involves elevated temperatures. Yaashikaa *et al.*, (2020)described how the charring process, influenced by temperature, can alter the surface morphology of biochar. Increasing the temperature during the charring process results in larger pores in the biochar, thereby enhancing its porosity (Yang *et al.*, 2021).

Meanwhile, in fluff biobriquettes, the pores become tighter and denser with the percentage of the C element increasing to 71.48% and the O element decreasing to 20.33%. The increasing density of pore size in biochar can result from various factors, including the incorporation of adhesives and the application of pressing pressure, which enhances the bond between biochar particles and adhesives. Furthermore, immersing biochar in cooking oil was an additional adhesive, effectively reducing the size of the briquette's pores. The addition of adhesives and immersion in used cooking oil are factors that can elevate the percentage of C elements in the sample. Elements including Mg, Al, Si, S, Ca, K, P, and Cl present in the tea fluffs, tea fluff biochar, and tea fluff bio-briquette samples were ash constituents. Test samples exhibiting a higher proportion of C and O relative to other elements suggest a low ash content (Hadey *et al.*, 2022)..

Table 10
EDX Analysis Results of Tea Fluffs, Tea Fluff Biochar and Tea Fluff Biobriquettes

Elements	Tea Fluffs Raw Materials (wt%)	Biochar Fluffs Tea (wt%)	Tea Fluffs Biobriquettes (wt%)
C	68.89	68.17	71.48
O	27.29	26.30	20.33
Mg	1.83	0.31	0.36
Al	0.14	1.10	0.82
Si	0.01	1.24	2.81
S	0.11	0.07	0.24
K	1.02	1.74	3.29
Ca	0.25	0.55	0.33
P	0.11	0.17	0.25
Cl	0.03	0.25	0.07
Total	100	100	100

Source: Research Data (2024)

4. Conclusion

Following pyrolysis, tea fluffs exhibit increased ash content, fixed carbon, calorific value, and reduced moisture content and volatile matter. The calorific value increased from 4,482.56 to 6,374.98 cal/g. The properties of tea fluff bio-briquettes adhere to SNI 01-6235-2000 regarding moisture content, ash content, and calorific value. Additionally, the parameters for volatile matter, fixed carbon, burning rate, density, and compressive strength conform to Japanese quality standards. ANOVA results indicate that biochar particle size, resin gum adhesive concentration, and immersion time in used cooking oil significantly affect each response, as evidenced by the resulting p-value in the model (p-value <0.05) and the lack of fit value (p-value >0.05). The BBD RSM analysis indicates that the optimal conditions for tea fluff bio-briquettes are a biochar particle size of 850 µm, a resin gum adhesive concentration of 11%, and an immersion time of 5 minutes in cooking oil. The duration of immersion of bio-briquettes in cooking oil is the primary factor influencing the quality of the tea fluff bio-briquettes, particularly regarding their calorific value. CHN, FTIR, and SEM measurements indicated increased carbon elements, supporting the feasibility of producing tea fluff bio-briquettes.

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