

Research Article

Tangibility of Design of Experiments on the Evaluation of Biofuel Briquettes Made from Rice Straw for Multiple Qualitative Parameters

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Abstract

This research aims to investigate the production of low-cost biofuel briquettes from rice straw using suitable binders in different ratios and to characterize rice straw-based biofuel briquettes produced by the mechanical press method. In this research, thirty different samples were taken in different combinations for rice straw and binding material, based on the variation of particle size, binding material concentration, and type. This study concluded that briquettes produced from rice straw of Particle Size (PS) 2 mm bonded by 30% paper pulp and rice straw of PS-5 mm bonded by 30% cow dung + paper pulp mixture have higher calorific value of (4253 kcal/kg), which was followed by treatments rice straw of PS-1mm bonded by 20% cow dung (4232 kcal/kg) > RS of PS - 1mm bonded by 20% CD > RS of PS- 5mm bonded by 30% PW (4210 kcal/kg) > RS of PS- 5mm bonded by 10% PW (4199 kcal/kg), which are quite higher than commercially available cow dung briquettes (3456 kcal/kg). They are also significantly more environmentally friendly than traditional firewood. Briquettes combust more effectively and cleanly than firewood because they are more energy-dense and often drier.

Keywords: Stubble burning; Eco-friendly; Binders; Cow dung; Paper pulp.

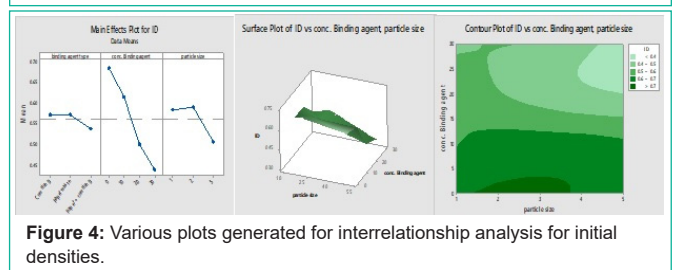
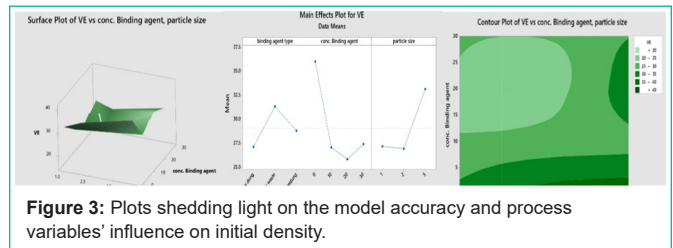
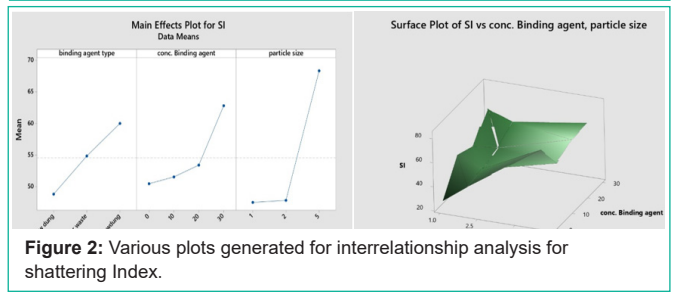
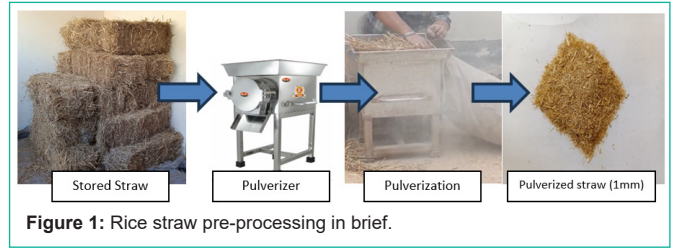
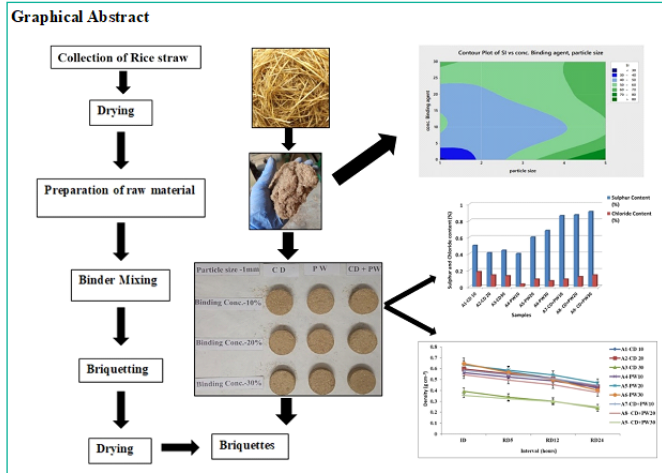
Introduction

The widespread use of commercial energy has improved the quality of life but has also brought about several environmental problems. Biomass is a significant renewable energy source, contributing around 10% of all energy [1,2]. Biomass is a fuel from organic waste produced by living things, such as plant, animal, and municipal waste [3]. However, most countries facing various environmental issues due to waste disposal and environmental pollution from burning agricultural residues lack strategies for managing solid waste [4]. Agro-waste residues are related to disposal and transportation in addition to this issue. The large volume of crop leftovers is handled more quickly and easily by burning, so farmers favor it. However, the smoke created by burning agricultural waste can still cause some respiratory diseases [5]. According to Bhattacharyya et al. [6] and Verma et al. [7], Punjab farmers burn 81% of rice straw in their fields annually in India. Farmers in Punjab and Haryana burn an estimated 35 MT of crop residue each year after harvesting their paddy fields [8]. Therefore, burning them outside causes significant environmental damage.

A better option for using those agricultural wastes is biomass gasification or densification. Compacting biomass material into a uniform briquette is known as densification or briquette [9]. Combustible biomass substances are compressed into a block called a briquette. It is a physical process. Strength, density, portability, and

the quantity of heat released per volume of biomass are all improved by briquetting [10]. Due to their high production costs, briquettes need a reliable market and a significant and consistent supply of acceptable raw materials to be successful [11]. Briquettes are used for cooking fuel, electricity generation, and heating, usually in developing countries that do not have access to more traditional fuel sources. Briquettes can be used in developed countries to produce electricity from steam power by heating water in boilers [12]. However, charcoal in briquettes has many drawbacks, including releasing greenhouse gases such as CO₂, SO₂, NO₂, and CH₄ [13].

Briquettes' low sulfate content, relative lack of dust, ease of handling, and high calorific value are among their most significant benefits. The use of binder material strengthens the briquettes. Inorganic or organic substances can act as binders. Among the recognized organic binders are molasses, starch, and heavy crude oil. Clay, sodium silicate, cement, and charcoal dust are inorganic binders [14]. It was reported that cow dung with different materials has a higher calorific value than starch as a binder [15], and adding waste from a paper mill increased the briquettes' shatter index [16]. Because it contains lignin, which helps to bind the particles together into the briquettes, paper waste is a valuable binder for briquetting. The raw material's moisture significantly impacts the lignin's softening



temperature. At 30% moisture (wet basis), it is roughly 90-100 °C; at 10% moisture (wet basis), it is approximately 130 °C. Lignin does not become softer at room temperature. Protein also functions as a binder in its plasticized state, which also requires processing at high temperatures. Therefore, an additional supply of binding agents is required for processing at ambient temperature [17]. The study also examined the quality analysis of various briquettes utilizing dry leaves, wheat straw, and sawdust as feedstocks. The proximate analysis method was used to explore these briquettes. Following that, results were compared with commercially available cow dung briquettes. The calorific value of the briquettes made with these feedstocks and cow dung as a binder was 5920.40 kCal/kg, which was greater than the calorific value of briquettes made with other binder materials such as paper pulp (5874 kCal/kg) and commercially available cow dung briquettes (3452.34 kCal/kg). Compared to conventional cow dung briquettes, other attributes such as the percentage of ash content, the percentage of sulphur and chloride content, and the percentage of volatile matter were all lower [18]. However, the efficient utilization of rice straw as a biofuel faces challenges due to its low energy density, high moisture content, and poor handling characteristics. To address these issues, producing biofuel briquettes using rice straw and suitable binders presents a promising solution. The mechanical press method has shown potential in producing compact, energy-dense briquettes that are easier to transport and store. Therefore, this study aims to explore the production of low-cost biofuel briquettes from rice straw by experimenting with different binder ratios. Additionally, the research seeks to characterize the physical and combustion properties of the produced briquettes to assess their suitability as a renewable energy source.

Materials and Methods

Pre - processing of Rice Straw and Binder Preparation

Dry rice straw was gathered from ICAR-IARI, New Delhi farms for this experiment. To increase pulverization efficiency and eliminate blockages and irregular particle sizes, rice straw was exposed to open sun drying for 2 days. Following a thorough investigation into particle sizes, the cutting was done in a commercial pulverizer with a 3 HP capacity made by Kalsi Enterprises into the several available particle sizes, namely 1 mm, 3 mm, and 5 mm, utilizing sieve-based attachments suitable with the machinery (Figure 1).

For maximum uncertainty, raw cow dung was gathered from nearby micro cow breeders and then kept in a cold, dry location. Preliminary investigation on cow dung revealed 40% of the moisture on dry ash weight basis prior to briquette production. Newspaper waste was purchased from nearby stores in a fresh slot, and it was brutally sliced into long strips. Following simulation and estimation of the highest quantity of paper needed, around 300 grams were submerged in 400 grams of effluent for two days. The strips were pulverized after they lost their structural strength in order to provide paper pulp waste for binding.

Modeling Experimental Runs and Binder-Incorporated Molding

This study investigated the briquetting behavior of rice straw using cow dung and paper waste as binders using the basic design of experiment protocols; the basic architecture was a multilevel factorial design to analyze the effects and mutual interactions of binding agent and particle size on the quality of the briquettes. The analysis was aimed to optimize the briquetting machine for 12 qualitative parameters Shattering Index (SI), Volume Expansion (VE), Density Ratio (DR), Initial Density (ID), Relax Density (RD), Compaction Ratio (CR), proximate analysis parameters, Ignition Time (IT),

Burning Time (BT) and Calorific Value (CV). The experiment explored the combined effects of three factors: rice straw mass (100, 72, 64, and 56 g), binder type (cow dung, paper waste, and cow dung + paper waste), and particle size (1, 2, and 5 mm), on the briquette properties. A total of 36 experimental combinations were generated following the RSM design, allowing for the evaluation of both individual factor effects and potential interactions between them. The subsequent analysis employed a general factorial regression model to quantify these relationships and establish a predictive model for briquette properties based on the chosen factors and their levels. All three particle sizes—1, 2, and 5 mm—went through this procedure, apart from the cow dung + paper waste scenario, where a 1:1 binder mixture was added. A total of 27 experimental run combinations were produced in this manner according to the general factorial design. Three sets were created with only rice straw for separately designing each of the three particle sizes (1, 2, and 5 mm) samples without binders. Following binder mixing, the mixture was fed into the briquette press and compressed for a minute.

The complete factorial design is an essential strategy for constructing experiments exploring several factors' influence on a single response variable. Researchers may explore individual main effects and potential interactions between factors since it systematically analyzes all possible factor value combinations. This all-encompassing technique makes understanding the system under investigation simpler, allowing researchers to identify the most significant aspects and how they interact to influence the response. Although it is beneficial for its simplicity and efficacy when there are few variables and when there are many factors, the number of runs required climbs exponentially, necessitating the adoption of other designs, such as fractional factorials. A regressive model was used to examine the main and interaction influences of three process factors on the 10 responses; this served as the foundation for establishing the briquetting trials. All factorial designs have three essential characteristics: estimable model terms, projection, and orthogonality. Using a factorial design, the experiment evaluates every possible level combination for each ingredient. The 30 experimental runs worksheet consists of different columns of process variables with combinations designed to investigate each main effect, each two-factor interaction effect, and each three-factor interaction effect because every combination of factor and level is included in the 2^k complete factorial design experiment (Table 1).

Minitab 17 was used to analyze and model response data using the response surface approach. A second-order polynomial model was fitted to the experimental data:

$$Y = \beta_0 + \sum_{i=1}^4 \beta_i X_i + \sum_{i=1}^4 \beta_{ii} X_i^2 + \sum_{i < j=1}^4 \beta_{ij} X_i X_j$$

Where X_i and X_j are the independent variables in coded values, β_0 is the constant, β_i , β_{ii} , and β_{ij} are the regression coefficients. Y represents the responses' calorific value, proximate analysis parameters, ignition time, SI = Shattering Index, VE = Volume

Table 1: Coded independent variables and the ranges used for the factorial design.

Variables	Range
Types of binding agent	Cow dung, paper waste, cow dung + paper waste
Concentration of binding agent	0, 10, 20 and 30
Particle size of rice straw	1, 2 and 5mm

Expansion, DR = Density Ratio, ID = Initial Density, RD5 = Relax Density (5hr), RD12 = Relax Density (12hr), RD24 = Relax Density (24hr), CR = Compaction Ratio. Analysis of Variance (ANOVA) was used to analyze the model. The lack-of-fit test was performed to assess the model's suitability, and the coefficient of determination, or R^2 , was computed. Response surfaces were created to investigate how interactions affect responses.

The briquette machine was a fully hydraulic prototype platform indigenously developed at the Centre of Excellence in Farm Machinery, CSIR-CMERI, Ludhiana, Punjab. It has a power input of 9HP, simultaneously producing at least 4 briquettes. During the operation of the briquette machine, the rice straw is mixed with 10% cow dung or any suitable binder, such as paper waste, along with water so that the consistency is similar to dough. The mixture is fed in the hopper conveyed by the auger to the exit tubes from where the cylindrical briquettes come out and fall on the pan placed under the machine. A manually operated biomass briquette machine was planned and constructed, which consists of 3 moulds with a diameter of 35mm each. Holes were provided to remove excess water. At the bottom, a 20 mm thick plate was provided with holes to hold the briquettes and remove the water. Each hole has a 3mm diameter. At the top of the machine, a screw is pressed; the plates move downward and press the feed. However, answering the inevitable question of biomass and binding agent eccentricity and optimization of process variables applicable to all briquetting machine types was the scope undertaken for this study.

Qualitative and Quantitative Analysis Parameters

A standard approach was used to analyze various quality characteristics, namely, calorific value [19], moisture content [20], ash content [21], volatile matter [22], fixed carbon [23], density [24], ignition time [25], burning time [26], shattering index [27], volume expansion [28], density ratio [28], relax density [29], and compaction ratio [30]. The data were analysed using the Minitab 17 software. The treatment means were compared using Critical Difference (CD) at a 5% ($p = 0.05$) level of significance.

Results and Discussion

Statistical model Based on General Factorial Regression

In this section, aided by analysis tools, we will investigate further the relationships obtained from the fitted experimental outputs. When ANOVA analysis was performed for 12 responses, the three-way interaction yielded no F-value, T-value, and P-value. This could be because statistically significant relations between all three variables were missing. Hence, a relationship was established considering only linear and two-way interactions for analysis. The following subsections will meticulously discuss each process variable's results.

Shattering Index

The plots below signify the process variables' influence on the briquette-shattering index (Figure 2). The main effects plot depicts a significant relationship between the binder types, and as first viewed, cow dung was the best-perceived binder degrading shattering index; nevertheless, the combined effects of both binders raised the shattering index due to binder immiscibility. The increased concentration of binders had a detrimental influence on the

performance, with finer particle sizes outperforming coarser particle briquettes, indicating improved adhesivity with finer particle sizes. The ANOVA analysis revealed that the concentration of the binding agent has a more substantial impact on the greatest F-value of 4.67, followed by particle size at 3.54. The combined effects of particle size and binding agent type are more significant, with bigger neighbouring mean squares and a higher F value of 1.37; the adjacent P-values were also lowest for these two variables, suggesting a better positive probabilistic stance. Our model successfully predicted the shattering index of the briquettes. This is evident in the high Coefficient of determination (R^2) value of 0.91, indicating a strong relationship between the chosen variables and the shattering index. The residual plots further confirm the model's adequacy. They show that the selected variables are sufficient for the studied ranges and that there is no significant influence from unconsidered variables. The normal distribution of residuals (confirmed by the normal probability plot) and their randomness throughout the plots (histogram, residual vs. fit, and residual vs. observation order) all suggest a good fit with minimal impact from outliers or hidden factors. The T-value for the model coefficients suggested that paper waste had a more positive influence. In contrast, cow dung interestingly had an adverse effect on the shattering index, which was also observed in the main effects plot. The T values also indicated that the particle size also has an adverse effect on the shattering index, improving the quality of the briquettes when finer material is chosen. The following are the indices for the briquettes under study, from lowest to highest, ranging from 22.21% for *C. gabunensis* to 99.16% for *C. pentandra*: *C. pentandra*, *A. toxicaria*, *C. gabunensis*, and mixed type. The low-density wood *C. pentandra* briquette has a higher shattering index than the mixed variety, indicating it is more resistant to gravitational deterioration. The *C. pentandra* chippings' higher porosity led to better compaction and harder briquettes than the composite and the other briquettes [31].

Volume Expansion

The various graphs deduced from the model shed ample light on the influence of variables over volume expansion (Figure 3). The chosen graphs and the observation are explained in this section. The surface plot of volume expansion clearly explains that adding the binding agent decreased the volume expansion. However, particle size also played a significant role in improving adhesion and degrading volume expansion. It is evident from the main effects plot of binding agent concentration vs. mean VE that adding a binding agent improved the quality of briquettes by adversely affecting volume expansion. We observed the main effects plot and concluded that cow dung was the best binding agent for improving volume expansion properties. The binding concentration with the most minor volume expansion belonged to 1 and 2 mm of particle size and was formed by adding binders between 10 to 30% by weight; the contour plot signifies an improved performance by decreased volume expansion in the samples mentioned above. In the corresponding ANOVA analysis, it was observed in linear interaction that the concentration of the binding agent had the most significant impact on the volume expansion from the highest F-value of 4.67. When mutual interaction was needed, the 2-way interaction of binding agent type and particle size played the most crucial role with an F-value of 1.37. Their respective P-values clocked the least up to 0.02 and 0.30. The model's

overall coefficient of determination (R^2) was 0.75, achieving a good fit and predictability accuracy. From the T-value test data results, it was evident that cow dung adversely affected VE, showing negative 1.29 and particle size decrease, and adding more binder generally degraded the volume expansion. Briquettes made from 2 mm of rice straw mixed with cow dung had the most significant effect, with the lowest negative value of -0.17, concluding a diminishing impact on volume expansion. This matches the fact that the improvement could be achieved when using these binders with lower particle sizes. The residual analysis complemented the accuracy of the prediction model and the interpretations. The normal plot of residual distribution confirms even scattering of the data by being centered on the line of predicted residuals running through the middle. From the observation of the histogram and fitted vs. residuals, it is inevitable that no correlated factors are left out as process variables due to their symmetric nature and lower residuals overall. The observation order vs. residuals further affirms that the randomness of the residuals deprives us of the fact that another variable has a more significant correlation with the volume expansion than the process variables. When briquettes of 6.5 and 2.5 mm particles were made in a small mould, the volume expansion was reduced by 16.4-30.1% and 31.3-60.2%, respectively [28].

Initial Density

According to the main effects plot, the average initial density falls as the particle size of the binder rises, contradicting prior findings. Cow dung was the most effective binder, with a greater beginning density (Figure 4). However, increasing the concentration of the binding agent led to a drop in initial density. This might be due to the volume of the briquette, including the original moisture content of the binder pulp and dung since the binder added wet weight; however, dry weight analysis was utilized to calculate the weight of the sample briquettes spanning moisture-free samples. This can be validated by looking at the surface and contour plots, which show that the initial density declines as the proportions of binding agents rise. The main effects plot shows that briquettes composed of a mix of both binders degrade the response initial density more. The normal probability plot indicates tightly packed and neatly ordered model residuals, with the lowest values occurring the most frequently, as shown in the residual histogram; this leads to the higher Coefficient of determination (R^2) values of 0.94 and a mean model error of just 0.032. The ANOVA results initially suggested that the most crucial factor influencing the binding agent concentration was with an F-value of 40.83 and a P-value of 0.003, it accurately described the notional impacts of moisture content in the initial density of the briquettes, including increased transportation costs and total initial mass loss to the beneficiaries. With an F-value of 2.96 and a P-value of 0.052, the two-way interaction between binder concentration and particle size had the greatest preferred influence on initial density. The other interaction has a p-value greater than 0.05 due to decreased weightage. The plot of residuals vs fitted values showed a random distribution, which explained the absence of observable elements that were not included in this experiment. The residual vs observational order graph demonstrates that randomness refers to a lack of relevant parameters for this present investigation, or that the examined parameters have an overpowering impact on unconsidered parameters, reducing their influence on the outcome.

Density Ratio

The propensity of unadulterated rice straw briquettes to bulge out and increase in volume was reduced when a binding agent was applied to the samples, enhancing adhesiveness (Figure 5). The contour plot indicated that low-density deterioration after 24 hours and taking up more space with increasing time were strongly influenced by the binder's improved storage capabilities. The surface plots supported that greater values were also recorded in lower particle sizes due to enhanced gumming from smaller particles and a minor amount of binding agent, which dramatically affected the density ratio. The ANOVA analysis revealed the same results: the linear effects of particle size were the most significant, with an F-value of 5.19 and a meagre P-value of 0.032, followed by the binding agent concentration at 5.19 and 0.016, respectively, as well as the 2-way interaction of binding agent concentration and particle size, which ranked first with the highest F-value of 2.62 and the lowest P-value of 0.088. The primary impact plot still shows cow dung as the best option for enhanced performance, but adding more than 20% binders appears unnecessary. The T-value supported this observation for cow dung, the highest at 1.98, followed by the 20% concentration of cow dung at 1.69, with matching P-values of 0.072. The whole model obtained a coefficient of determination (R^2) of 0.80, indicating high accuracy.

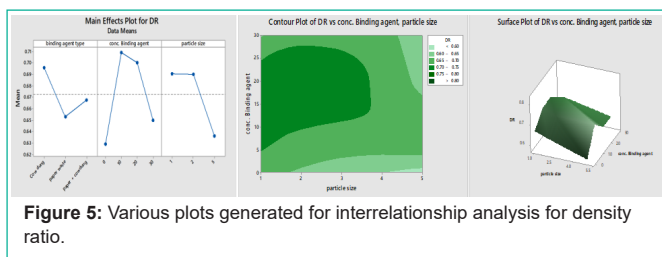


Figure 5: Various plots generated for interrelationship analysis for density ratio.

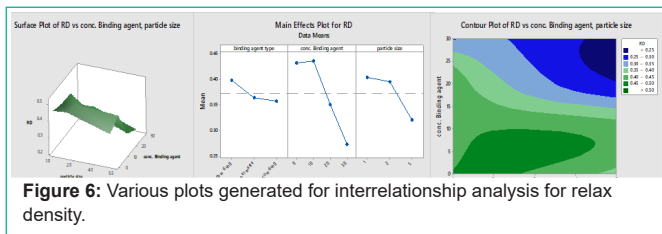


Figure 6: Various plots generated for interrelationship analysis for relax density.

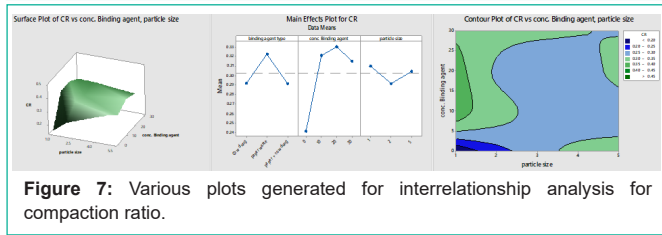


Figure 7: Various plots generated for interrelationship analysis for compaction ratio.

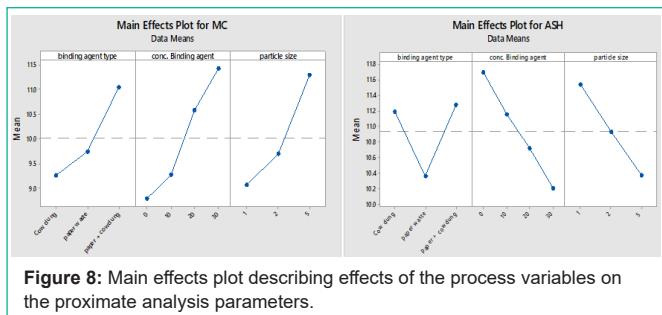


Figure 8: Main effects plot describing effects of the process variables on the proximate analysis parameters.

The residual analysis displays a descriptive normal probability curve, which explains negligible skewness and considerable fitness with fewer outliers. The Residual versus Fits plot also reveals that the fitted values are randomly organized, indicating a lack of unconsidered important parameters combined with the unpredictability of the observational order graph, implying increased model fitness for density ratio responses. However, the histogram plots contradict the preceding finding since residuals with the same tiny size but different signs appear the same number of times, implying a slight effect of an unconsidered variable. The result shows that tapped bulk density improvement of larger particles is higher, as also observed by Gomes et al. [32] for wheat straw and switch grass. When tapped, the density of loose and knife-milled rice straw particles did not change. This is because there is less particle contact surface area and more space, which causes tapped particles to emerge from the box rather than settle.

Relax Density

The relax density of the briquette is important in determining the storage qualities of the biomass, and the graphs show the decrease in density caused by mass loss after briquette storage (Figure 6). It positively detects and represents the mass loss in the main effects plot of concentration, with larger particles losing more moisture during storage. The contour map with the greatest densities at lower concentrations depicts packing inefficiencies, and an increase in particle size reduces the relax density, as shown by the surface plot. When ANOVA analysis was performed, an uncanny similar resemblance of binding agent concentration greatly affected the response, clocking an F-value of 25.28 with a corresponding P-value of 0.001 in linear interactions and particle size of rice straw and the concentration of binding agent clocking the largest F-value at 3.67 with a P-value of 0.036, indicating a better fit. It also implies that moisture has a significant influence on briquette characteristics. The model's coefficient of determination (R^2) was 0.92, indicating a solid match for predicting the changes expected to be measured. The normal distribution and frequency plots indicated that improved accuracy was achieved by deducing a high probability of low-value residuals occurring more frequently and a meager chance of positive residuals explaining the absence of high-value misfits. Residual vs. observation order and residual vs. fits yielded the same result: random residuals with random and widely distributed arrangements, implying no fixed structure. Hence, a lack of identifiable and influential variables was concluded from these two plots. Cow dung here, too, was the best-considered binder from the main effects plot and had the highest value among the binder types at 2.28 and a p-value of 0.042. The combined effect of cow dung and 2 mm particle size clocked the highest T-value of 3.13 at a P-value of 0.009. The binding agent at 20% seconded with a particle size of 1mm of rice straw was seconded from the interaction plot, contradicting the above statement. However, this could be because the increased particle size had a more considerable moisture loss. The compaction process can be an alternative for improving the energy density of these wastes [33]. Due to the elastic nature of biomass materials, the initial density of the briquette is higher when it exits the compression chamber and decreases over time. The size of the biomass particles, the compaction pressure, the briquetting temperature, and the briquetting time all affect the density of the briquettes [34]. Factors about the raw material and the

production method may impact the characteristics and quality of the solid biofuel. To increase production and raise the caliber of the finished product, it is possible to modify the particle size, compression temperature, retention time, and pressure. Particle size is one of these variables that matters the most since the lignocellulose materials lignin-lignocellulose bond is a reliable sign of the briquette quality [35].

Compaction Ratio

The following figures, created from the regression model, suggest an overall rise in the compaction ratio when a binding agent is added. The surface plot shows that decreasing particle size and rising binding agent concentrations increased the compaction ratio; the contour plot demonstrates the same connection (Figure 7). As predicted, the primary effect plot shows a significant rise when binders are placed into the briquettes. However, as the main effects plot shows, paper pulp outperformed other binding agents. According to the contour plot, the smallest particle size briquettes with 15 to 20% binder adulteration achieved the highest compaction ratio. The regression model's ANOVA analysis yielded a coefficient of fitness (R^2) of 0.78, indicating that the model is well-fitted. Linear interaction revealed that the concentration of the binding agent had the most significant impact on the response, with a maximum F-value of 3.70 and a corresponding P-value of 0.043. Like other physical characteristics, the two-way interactions were primarily influenced by the combination of binding agent concentration and particle size, as shown by the greatest F-value of 3.40 and a corresponding P-value of 0.034. The normal probability plot of the residuals revealed a skewed line with short tails, indicating that tiny outliers in the design reduced the fitness value. Paper waste was identified as the best binder positively affecting compaction, with a subsequent T-value of 1.37, particle size of 1 mm, and binding concentrations of 10%, resulting in the best compaction with a T-value of 1.58. Overall, adding the binding agent was helpful since the absence of a binder had a detrimental influence on compaction, as did all the virgin rice straw briquettes, resulting in a negative T-value. Additionally, the compaction ratio values found in this study are comparable to the well-known biomass residues. For example, when rice husk was briquette, a compaction ratio 3.80 was attained, whereas groundnut and melon shells yielded compaction ratios of 4.2 and 3.5, respectively. Similarly, Boluwafi's briquetting of guinea corn residue resulted in a compaction ratio of 3.20 to 9.70 [36].

Proximate Analysis of Briquettes

Main effects plot: As anticipated, the moisture content increased with the binder concentrations and the combination of cow dung and paper waste. Particle size increases added to the moisture content due to higher moisture loss due to larger particle sizes. The ash content was significantly affected by recording diminishing ash content with an increase in the binder content due to the lower ash content of the binders than rice straw (Figure 8). The particle size also negatively affects the ash content, as anticipated by the plots. This could be because ash could convert to particulate matter at lower sizes and vented off from the furnace. The volatile matter concentrations were negatively impacted by increased binder concentrations and particle sizes. Combining both binders gave the most promising results of low moisture content and lower ash content with high fixed carbon and volatiles. As a result, one drawback of employing rice husks is that

they have a high silica concentration, which causes combustion to produce a lot of ashes, which can cause operational issues like slag and clogging [37].

Analysis of the contour plots reveals a positive association between binder concentration and briquette moisture content. Conversely, surface plots indicate a negative association between particle size and moisture content (Figure 9). Additionally, the surface plots demonstrate a significant interaction effect between binder concentration and particle size on ash content. The combination of larger particle size and increased binder concentration substantially reduces ash percentage from exceeding 13% to less than 9%. The ash content contour plot also identifies a cluster of samples with an average ash content ranging from 10% to 11%. These samples have particle sizes between 2mm and 4mm and exhibit a wide range of binder concentrations, suggesting no significant independent effect of binder concentration on ash content within this specific particle size range.

ANOVA and Residual Analysis of Proximate Analysis Parameters

This analysis investigates how different factors influence the properties of briquettes, explicitly focusing on moisture and ash content. ANOVA analysis for the regression model revealed that

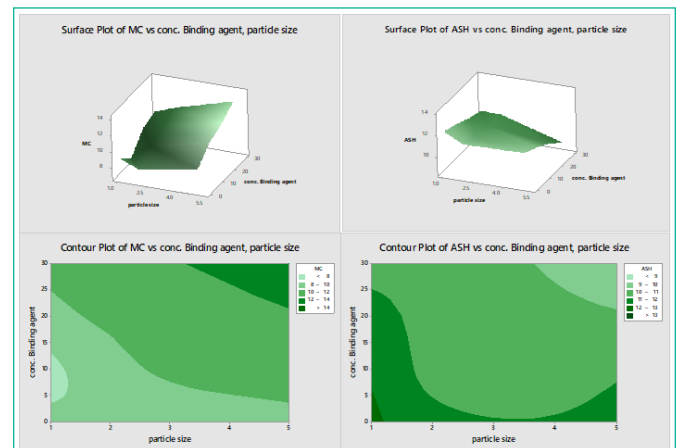


Figure 9: Contour and surface plots of the process variables on the proximate analysis parameters.

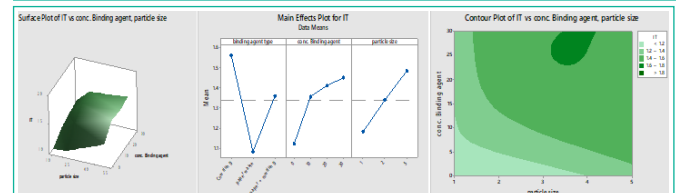


Figure 10: Various plots generated for interrelationship analysis for ignition time.

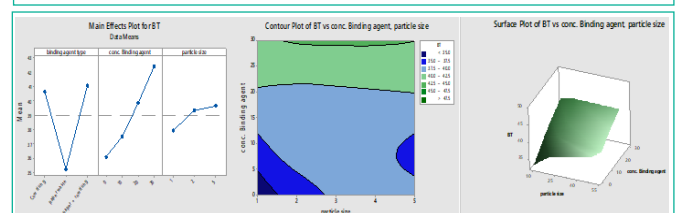


Figure 11: Residual plot of Burning Time signifying influence on residuals.

the model significantly explains a high proportion of the variance in moisture content ($R^2 = 87.87\%$). Briquette moisture content increases with increasing concentration of binding agent. Smaller particle size is associated with lower moisture content in the briquettes. Briquettes made with paper waste generally have lower ash content than those made with cow dung or a combination. Smaller particle size (1 mm) leads to higher ash content than larger particle sizes (2 mm and 5 mm). The model significantly explains a high proportion of the variance in ash content ($R^2 = 88.65\%$). All main effects (binding agent type, concentration, and particle size) have a significant impact on ash content (p -values < 0.05), out of which particle size and concentration of binding agent improved has the highest recorded F -value of 8.63 and 7.63, respectively, followed by binder agent at 6.51. The interaction effect between binding agent type and particle size is exciting. For cow dung, briquettes with a 1 mm particle size have the highest ash content, while those with a 2 mm size have a lower content. The trend is reversed for paper waste, with 1 mm particles having lower ash content than 2 mm particle-sized straw briquettes. The VIF (Variance Inflation Factor) values for all terms are close to 1, indicating no significant multicollinearity among the independent variables. A statistically significant interaction effect (p -value < 0.005) was observed between binding agent type and particle size for ash content. This indicates a non-additive relationship, where the combined influence of these factors on ash content depends on the specific combination used. Notably, the F -value of 6.50 associated with this interaction effect is the highest among all two-way interactions, highlighting its relative importance.

Furthermore, analysis of the volatile matter content plots suggests an inverse relationship with binder concentration. Increasing binder concentration appears to be associated with decreased volatile matter content. This could be attributed to the loss of volatile components during briquette formation processes, potentially due to pressure, heating, or the observed decrease in ash content. The reduction in ash content might be linked to an increase in fixed carbon content, as evidenced by the rise from less than 10% to over 30%. The R square for VM and FC is 0.94, respectively. The fits emerge a better understanding of the characteristic effects of binder on the combustibility of the briquettes and how high-grade energy source it is.

Ignition Time

The plots provide a positive correlation between binder concentration and particle size with the ignition time. This behaviour was discreetly notable from the surface plot, showing a steep increase in the ignition time with subsequent elevated binder concentrations. The increase in particle size elevated the ignition time, too, as interpreted from the main effects plot of particle

size vs. mean IT (Figure 10). The ignition time peaked at higher concentrations of binding agents and longer particle sizes on the contour plot. Paper waste performed the best, decreasing the ignition time to lower than the rice straw itself, suggesting better performance; ANOVA analysis supports this observation; the binding agent type has the highest recorded F -value of 153.80 with a corresponding P -value of 0.00 confirming a major significant influence. Longer ignition time translates to a slower initial burning rate. This means it takes longer for the briquette to reach its peak heat output, reducing its efficiency in applications where rapid heating is desired (like fireplaces or

boilers). The regression model's overall coefficient of determination (R^2) was recorded at 0.98, implying premium fitness. The two-way interaction calculating ANOVA analysis resulted in the combined effect of binding agent type and the binding agent concentration as the most significant influence on the ignition time, clocking the largest F -value of 19.61 and corresponding P -value of 0.005. The ANOVA analysis also reconfirmed the observation of paper waste being the most influential parameter in decreasing ignition time, with its largest negative T -value of -15.91. Paper waste at 20% concentration inlaid the most significant combined negative influence with a T -value of -2.76. The residual analysis sheds some critical light on the model's fitness, implying whether the deductions are chance-based random observations or a significant influence is being recorded. The normal probability plot concludes that no significant or large outliers are neatly arranged over the mean fitted line, indicating no large residuals are fitted. The fitted value vs. residuals confirms that the fitted values were purely random and did not follow a pattern. The histogram predicts only lower magnitude residuals are left, explaining better accuracy and fitness. The observation order vs residuals also claim the same results: that the residuals are random; hence, outliers are minimal, and unconsidered variables have a much lower influence than the variables used in this study. Fuels with a high volatile matter content typically ignite faster. A briquette with a long ignition time might suggest a lower volatile matter content, which could also impact the burning rate and energy release. High moisture content can hinder ignition and slow down the burning process. However, excessively dry briquettes might ignite too quickly and burn out rapidly. A slower burning rate also translates to a lower rate of energy release. This means the briquette might not be able to provide sufficient heat for the intended application over a desired timeframe. To use charcoal briquettes as fuel more effectively, it is crucial to understand the elements that influence the pace of burning and the time it takes for the briquettes to ignite [38]. Briquette density affects how quickly a flame spreads since there is less room for mass diffusion due to its low porosity, which prevents drying, depolarization, and burning [39].

Burning Time

The burning time of fuel, such as rice straw-based briquettes, is critical since it profoundly impacts efficiency, cost-effectiveness, environmental impact, and practicality. A longer burning span implies the fuel is consumed more slowly, resulting in greater efficiency, especially in applications that need sustained energy release. This delayed consumption reduces fuel costs since less fuel is required to produce the same energy. Regarding environmental impact, longer burning times can result in more complete combustion, minimizing harmful material emissions and creating a cleaner, healthier atmosphere. As seen in the image above, cow dung performed better than paper trash, and the combination had the most extended burning period (Figure 11). The burning time rose as particle size improved, as seen in the main effect plot; nevertheless, the combined effects of binding agent concentration and particle size from the surface plot indicate that the binding agent addition enhanced burning time. As particle size grows from 3 to 5, so does the burning duration at all concentrations of the binding agent. This indicates that bigger particles take longer to burn. At lower concentrations of the binding agent (around 20), there are regions where the burning time is significantly higher (dark green areas indicating a burning time

greater than 45 seconds). Conversely, at higher concentrations of the binding agent (around 30), there are regions with lower burning times (dark blue areas indicating a burning time between 35-37.5 seconds). This suggests that a decrease in the concentration of the binding agent leads to longer burning times. The contour plot signifies that the binding agent addition had more influence than particle size, as the highest burning time was observed with higher binder concentrations for all particle sizes. From the perspective of practicality, fuels with more extended burn periods necessitate less frequent reloading, which may be a substantial advantage in many applications, such as heating houses or powering industrial operations. Understanding and controlling the burning period of biomass briquettes is critical for realizing their full potential as a sustainable and efficient energy source. This insight enables the adjustment of factors such as particle size and binding agent concentration, resulting in improved briquette performance as a fuel source. However, the burning periods of the briquettes rose when more paper pulp and wheat straw were added as a binder [40].

ANOVA analysis on the regression model complements the above deductions mathematically; binding agent type had a significant influence. The linear interaction recorded the highest F-value of 132 with a corresponding P-value of 0.001, confirming a discreet influence on the burning time. The two-way interaction also recorded the highest F-value for the interaction of both binding agent type and concentration at 19.28. The combined effects of binding agent type and particle size had the lowest F-value, suggesting that the mutual exclusivity of both variables influenced the burning time. The regression model's overall coefficient of determination (R^2) was recorded at 0.98, implying premium fitness. The base level of burning time is 38.974 seconds when all other factors are at their reference levels. Using cow dung as a binding agent increases the burning time by 1.647 seconds compared to the reference level. Using paper waste decreases the burning time by 3.716 seconds compared to the reference level. A 20% concentration increases burning time by 0.869 seconds. A particle size of 1 decrease burning time by 1.047 seconds. A particle size of 2 increases burning time by 0.368 seconds. The P-values indicate whether the coefficients are statistically significant (typically, a p-value below 0.05 is considered significant). The VIF (Variance Inflation Factor) values suggest no significant multicollinearity among the predictors, with all VIFs being reasonably low (generally, a VIF above 5 or 10 is a cause for concern). For 20% binder and 1mm particle size, the T-value is 2.48, and the P-value is 0.029, which is significant. This suggests that a 20% concentration with particle size 1 increases burning time. For Paper waste 20, the T-value is -3.76, and the P-value is 0.003, which is significant. This indicates that a 20% concentration of paper waste significantly decreases burning time. Overall, the type of binding agent and its concentration significantly impacts burning time, with cow dung generally increasing it and paper waste decreasing it. The concentration of the binding agent also plays a role, with higher concentrations tending to increase burning time. Particle size has a less apparent effect, with some indications that smaller particles may decrease burning time. Interaction effects also show that one factor's impact can depend on another's level. The size of the biomass particles, the compaction pressure, the briquetting temperature, and the briquetting time all affect the density of the briquettes [34].

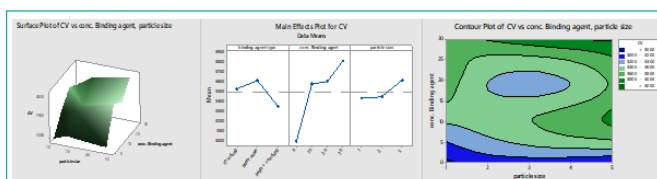


Figure 12: Various plots generated for interrelationship analysis for Calorific value.

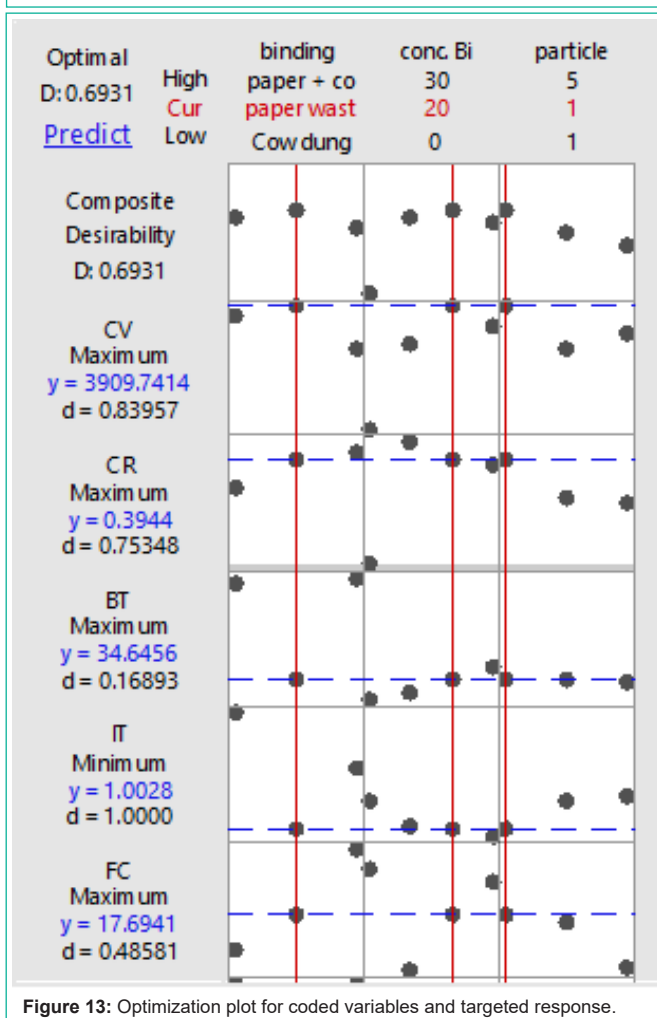


Figure 13: Optimization plot for coded variables and targeted response.

Calorific Value

A fuel's Calorific Value (CV) is one of the most critical aspects often used for qualitative analysis. Determining high-grade energy stored in biomass is important for designing reactors suitable for burning specific fuels. As we can see from the surface plot, the addition of binders significantly influenced the calorific value. The main effects plot signifies the steep increase in the calorific value due to the doped briquettes detecting an average rise of 600 cal/gm with just 10% of binder addition solidifying binder addition (Figure 12). The plot indicates that one type of binding agent results in a higher calorific value than the other. This suggests that the binding agent's chemical composition or energy content influences the briquettes' overall energy output. The size of the particles also affects the calorific value. Larger particle sizes are associated with a higher calorific value. This might be related to the surface area available for combustion;

larger particles may provide a more controlled burn, resulting in a more complete and efficient release of energy. The contour plot also explains the same hypothesis, denoting increased calorific value when added binders. Paper waste is the most influential binder, significantly adding to the calorific value as seen in the main effects plot. The ANOVA analysis concluded that a coefficient of determination of 0.97 explained a premium fit for the regression model. The overall model was highly significant owing to a P-value < 0.001, indicating that at least one of the factors significantly affects the calorific value. The most influential factor was binding agent concentration, sustaining the highest F-value at 104.40 with a P-value < 0.001, indicating a strong influence on CV. With an F-value of 21.00 and a p-value less than 0.001, the type of binding agent used is also a significant factor affecting the CV. Different binding agents likely have other properties influencing how much energy the briquettes can release upon burning. The particle size has the most negligible linear interaction on the CV, with the lowest F-value. In summary, the concentration of the binding agent and its interaction with particle size are the most significant factors affecting the calorific value of the briquettes. To improve the calorific value, one should focus on optimizing these factors. The 2-way interaction was also significant for the model with a P-value of 0.005. The combined effects of binding agent concentration and particle size significantly interact with the response, recording a significant p-value of 0.001. The non-significant interactions suggest that the impact of binding agent type and particle size are independent of each other in terms of their effect on calorific value. The normal probability plot of residual supports the model fitness as the residuals are normally distributed, indicating that model errors are unbiased and that the normality criteria of the regression have been met. In the residuals vs. fit plot, the residuals are randomly scattered around the horizontal axis without any discernible pattern, which indicates that the model has constant variance (homoscedasticity) and that the fitted values are unbiased. A lack of pattern or trend in the residual vs. order plot suggests that the residuals are independent and that no autocorrelation affects the model.

Response Optimization

During observation, a response optimizer tool was used to achieve and predict the best configuration of the process variables. It was configured based on the targeted values and goals to minimize and maximize the responses; a high calorific value was desired for better efficiency and performance; hence, it was targeted to be maximized. Subsequently, the compaction ratio was maximized as it indicates a denser briquette, which is less likely to break apart and can burn longer. It reflects the effectiveness of the binding agent and the pressure applied during the briquetting process. Density Ratio (DR) was targeted to be maximized. Higher DR suggests that the briquette maintains its shape and does not expand excessively after removal from the press, which is essential for handling, storage, and transportation. Volume expansion was minimized, and relaxed density was maximized as lower volume expansion is preferable as it signifies that the briquette will not disintegrate or become too fragile upon drying. A higher RD indicates a more stable briquette that will likely hold together better during handling and burning. Lastly, the shattering index was also minimized, preferably since a lower SI means the briquette is more durable and less likely to produce fines and dust during transportation and handling, which can be a loss of biomass

material and a potential safety hazard. Similarly, burning time was also maximized as it indicates that the briquette provides a sustained heat source, making it more efficient and economical for heating and cooking. It also suggests that less frequent refuelling is needed, which is convenient for the user. Subsequently, ignition time was also minimized because a shorter ignition time is desirable as it means the briquette is easy to light, which enhances the user experience. It also implies that the briquette can reach the desired temperature quickly, making it more practical for immediate heating needs. Proximate analysis parameters are also discussed; the moisture content was to be minimized along with the ash content as these adversely affect the burning efficiency, and more matter is needed to handle post and pre-power generation. Volatile matter content and fixed carbon content were also maximized due to some perks on combustion characteristics. For example, a higher fixed carbon content can lead to a longer burning time, while the presence of volatile matter can reduce the ignition time. Optimizing these parameters is crucial for creating high-quality briquettes that are user-friendly and meet the energy requirements efficiently.

The response optimizer's output for the briquette parameters indicates that the solution paper waste, with a binder concentration of 20% and particle size of 1 mm, has yielded results within the desired target ranges for most parameters. The Calorific Value (CV) of 3909.74 with a Standard Error (SE) of 83.6 is well within the maximum target range of 2947.91 to 4093.54, suggesting a high energy content beneficial for the combustion process. The 95% Confidence Interval (CI) and Prediction Interval (PI) indicate that the accurate CV will likely fall between 3727.5 and 4092, and future observations will likely fall between 3621.6 and 4197.8. The Compaction Ratio (CR) at 0.3944 with an SE of 0.0521 indicates a good level of compaction, and burning time (BT) at 34.646 with an SE of 0.793 is also within their respective maximum target ranges, indicating a compact and durable briquette that burns efficiently. The intervals show a consistent compaction ratio with a slight variation in future predictions. The Ignition Time (IT) of 1.0028 with an SE of 0.0547 is below the maximum target, which is advantageous as it implies a quick ignition. The intervals confirm this result's consistency, being between CI at 0.883 and 1.1219 and PI at 0.8144 and 1.1911. The Fixed Carbon (FC) content at 17.69 with an SE of 2.22 is within the maximum target range, contributing to the briquette's overall energy value. However, the CI falling between 12.85 and 22.53, whereas the PI ranging between 10.04 and 25.35, suggests that while the prediction is reliable, there may be more variation in future observations. The Ash Content (ASH) at 9.525 with an SE of 0.562 indicates a moderate level of ash produced, which is within acceptable limits as it suggests a lower tendency for clinker formation. The intervals show a moderate range of variation, which is 2% for CI and 3% for PI. The moisture content at 7.89 is within their minimum target ranges with an SE of 1.01, which is desirable for a stable burning process. The intervals indicate a moderate level of uncertainty in the prediction. The Volatile Matter (VM) content at 64.89 with an SE of 0.562 is within the maximum target range, which is crucial for the initial combustion phase. However, the CI ranges from 59.42 to 70.37, whereas the PI lying between 56.23 and 73.55 suggests a higher variation in future observations. The Relax Density (RD) at 0.4770, Initial Density (ID) at 0.6357, and Density Ratio (DR) at 0.7386 are all within their respective target ranges, indicating a good structural

integrity of the briquette. These densities are within the desired ranges with relatively low SEs of 0.037, 0.042, and 0.041, respectively, indicating good structural integrity and consistent quality of the briquettes. The Volume Expansion (VE) at 24.65 with an SE of 5.28 and Shattering Index (SI) at 43.10 with an SE of 7.02 is within their minimum target ranges, suggesting the briquette maintains its shape during burning and has a low tendency to break apart. The intervals, however, suggest a higher variability in these responses. Overall, the composite solution desirability of 0.631611 reflects a satisfactory optimization of the briquette parameters, with most parameters meeting or exceeding their target values. This indicates that using paper waste as a binding agent for rice straw briquettes could produce quality briquettes with good combustion properties. The results from this optimization can contribute significantly to the discussion on the feasibility and effectiveness of using agricultural waste materials as briquettes for energy production. The prediction intervals suggest that while the model is reliable, there is some variability in the responses, which should be considered when applying these results in practice. This analysis can be used as part of the results and discussion section in a briquetting paper for rice straw. Brand et al. [41] obtained the gross calorific value of 9.66 MJ.kg⁻¹ for briquettes manufactured with rice husk ash, which is lower than the values of the briquettes that include rice husk ash in the combination and 16.87 MJ.kg⁻¹ for briquettes formed of the mixture of rice husk and rice bran.

Conclusion

The data supports using agricultural waste, specifically rice straw and paper waste, as a sustainable and effective biofuel source. The comprehensive analysis of rice straw briquettes using paper waste as a binding agent demonstrates a successful optimization of key critical parameters for efficient fuel production. The ash content, volatile matter, and moisture content are at acceptable levels, ensuring minimal residue and efficient burning. The calorific value, compaction ratio, and burning time are within optimal ranges, indicating that the briquettes have high energy content, are well-compacted, and have a prolonged burning duration. As noted in the relax density, initial density, and density ratio, the structural integrity is robust, with the briquettes maintaining their shape and density throughout handling and burning. The ignition time and fixed carbon content further suggest that these briquettes are quick to light and maintain stable combustion. The low volume expansion and shattering index also point towards a durable briquette that resists disintegration and breakage. This approach provides a renewable energy solution and contributes to waste management and environmental conservation.

Highlights

Production of cost-effective briquettes from rice straw using various types of binders.

The briquettes achieved calorific values up to 4253 kcal/kg, surpassing commercial alternatives.

The briquettes combust cleaner and more efficiently than traditional firewood.

Multiple binder types and concentrations were explored to optimize briquette performance.

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Competing Interests

The authors have no relevant financial or non-financial interests to disclose and declare no competing interests.

Author Contributions

All authors contributed to the study conception and design. Material preparation, data collection and analysis were performed by [Lokesh Kumar Meena], [Renu Singh], [Sayon Chakravarty], [Madhuka Roy], [Brij Kishore], [Krishnendu Kundu], [Pooja LR], [Sibananda Darjee] [Bharti Rohatgi] and [Manoj Shrivastava] The first draft of the manuscript was written by [Renu Singh] and all authors commented on previous versions of the manuscript. All authors read and approved the final manuscript.

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