Quality Characterization and Manufacturing Process Optimization of Corncob Briquettes

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Abstract
Optimization of the densification process of agricultural residues is important to produce high-quality briquettes. In this study, the physical and mechanical characteristics of corncob briquettes were investigated to determine the influence of densification variables. The significance of the die temperature, compaction pressure, hold time and particle size on the density, water resistance index (WRI), lateral compressive strength (LCS) and impact resistance index (IRI) were determined using response surface methodology. Corncob briquettes had densities that ranged from 400 to 770 kg/m^3 whilst the WRI had values between 1.13 and 66.47%. The LCS ranged from 0.05 to 2.39 MPa, and IRI was between 5 and 200. The particle size has significant effect on all briquette properties. The pressure is also significant to the density and the LCS whilst the hold time is significant to the WRI and LCS. The compaction pressure interacted with particle size to significantly affect the LCS and IRI. From multi-response optimization, the optimum densification variables were 14.1 MPa, 132.9 °C and 15 minutes for compaction pressure, die temperature, and hold time, respectively, with particle size lower than 2.36 mm. Optimum densification variables resulted in density, WRI, LCS and IRI of 755.4 kg/m^3, 26.4%, 2.37 MPa and 169, respectively.

Keywords: Biomass densification variable, Box-Behnken experimental design, corncob briquette, response surface methodology, multi-response optimization, quality characteristics.

1. Introduction

Energy is a very important factor to be considered for driving technology, and the sustainable and speedy technological and industrial development is dependent on the regular generation and effective energy supply (Aremu, 2019). Energy generation using conventional sources has raised major concerns globally due to the large quantity of greenhouse gas emitted which results in global warming. Alternative energy sources are being exploited to slow down global warming. Biomass is an alternative energy source which is sustainable and renewable. Bioenergy has the potential to effectively replace fossil fuel using current technologies with minimal modification (Gustavsson et al., 2015; Reid et al., 2020). Numerous studies identify the huge global and regional potential of energy generation from biomass (Jekayinfa et al., 2020; Avcıoğlu et al., 2019; Ojolo and Orisaleye, 2012b; Tye et al., 2016).

Biomass has been used extensively for the generation of domestic energy using different developed cookstove technologies (Sutar et al., 2015; Ojolo et al., 2012; Orisaleye et al., 2018a). Biomass also has a very good potential for generation of industrial energy and electricity (Kumar et al., 2015). This can be achieved through the utilization of established technologies such as gasification (Situmorang et al., 2020; Ojolo and Orisaleye, 2012a), pyrolytic processes (Lam et al., 2016; Proskurina et al., 2017; Orisaleye et al., 2022c), fermentation (Kennes et al., 2016; Jimenez-Quero et al., 2017), biodiesel (Onukwuli et al., 2017; Ong et al., 2021; Bharadwaj et al., 2024) and biodigestion (Ona et al., 2022; Onofre et al., 2015). According to Basak et al. (2023), several emerging single-step, multi-step, and/or combined methods have been developed with the aim of achieving operationally simpler, economic and environmentally friendly alternatives.

In its raw form, biomass is often not usable in some of the energy conversion processes. This is because it is hygroscopic and has low bulk density, low energy density, and can rot during storage. Akhator et al. (2019), Ojolo and Orisaleye (2010), Kumararaja (2009) noticed that fluffy biomass with low bulk density caused problems during the operation of biomass gasifiers. Due to the bulkiness of raw biomass, it is often difficult to handle and expensive to transport. To address the problems caused by utilization of raw biomass, they could be transformed using densification technologies into forms that have better characteristics.

Densification technologies can be categorized based on the operating pressure (Oladeji, 2015). Low pressure technologies are produced with pressures less than 5 MPa and require a binder. Medium pressure technologies occur within the range of 5 and 100 MPa whilst high pressure technologies produce densified biomass at pressures above 100 MPa. Densification equipment using high pressure include pelleting machines, screw extruder briquetting machines, mechanical and hydraulic piston press technologies, and roller press technologies. The products of biomass densification are pellets, cubes and briquettes which are classified based on size and shape.

Some studies have been conducted using different technologies to assess the role densification variables in determining the quality of compacted biomass. Material variables include particle geometry, moisture content and biochemical composition. Process variables are compaction pressure, die temperature and hold time. Studies have also investigated the effects of additives and binders, and machine specific parameters such as speed, feeding rate, die size and so on. Other studies have investigated various biomass pre-treatment methods and how they affect the quality of briquettes. Orhevba et al. (2016) demonstrated that composite briquettes can be effectively produced from corncob and rice husk.

Zhang et al. (2019) also utilized Response Surface Methodology (RSM) to study the influence of densification variables on durability, density and impact resistance of briquettes produced from millet bran. Increasing pressure caused initial increase of density and durability before an eventual decrease. The impact of briquetting variables on the density of corncob briquettes were investigated by Orisaleye et al. (2018b) using a factorial design. Positive effects on the briquette density were observed with increasing compaction pressure and die temperature and decreasing particle size. Orisaleye et al. (2019) considered the water resistance of briquettes from corncob and observed that there was improvement of the property with higher die temperature and hold time, but with reduced particle size. Abineno et al. (2025) investigated the effect of particle size on physical and thermal properties of Kesambi wood briquettes and found that higher density and compressive strength along with enhanced combustion efficiency were achieved. Kuerban (2025) investigated the briquetting of cotton stalk and walnut shell and their blend considering different particle sizes. The study recommended briquetting of coarse cotton stalk and medium walnut shell particles in a ratio of 7:3 at 100 °C for residential and industrial use due to the suitability for fully utilizing local agricultural waste.

Muazu and Stegemann (2015) showed that starch and water addition improved the strength of briquettes but resulted in the reduction of the green and relaxed densities. Sekyere et al. (2025) examined the fuel properties of briquettes produced from sawdust, cocoa shell, corncob, palm kernel shell and coconut husk with 50% binder ratio. The study found that the CO_2 emissions from the briquettes were not significant enough to contribute to global warming when compared with the recommended ambient standard. Also, the CO emissions posed no health or environmental concerns as they were lower than the air quality guideline values. Ajagbe et al. (2025) investigated the quality of briquettes from carbonized cassava peel and sawdust and found that the ratio of the mixture of the materials was the most significant influence factor on the physicochemical and combustion propertie of the briquettes. The study carried out by Oyebamiji et al. (2025) showed that there was a high potential to produce efficient and sustainable briquettes produced from biochars of corncob, groundnut shell, rice bran, sawdust, corn straw, dry leaf, and sugar cane peel. Piarpuezán Enríquez et al. (2025) also produced briquettes from carbonized potato stalk using potato and achira binders. It was discovered that the calorific values exceeded the threshold of 12 MJ for biomass fuel classification.

Using factorial design, regression models were developed by Jekayinfa et al. (2019) to predict some physical properties of corncob briquettes whilst Niño et al. (2020) investigated mechanical properties of composite briquettes of rice husk and pine sawdust. The studies found that the briquette density and elastic modulus was significantly dependent on the hold time. The production of poplar briquettes using a hydraulic piston press briquetting machine was studied by Orisaleye et al. (2023). Die temperature was determined to be statistically significant to density and water resistance of poplar briquettes. Using a central composite design and RSM, Mainkaew et al. (2023) identified the parameters that optimize briquettes from elephant dung using cassava starch as binder and using a low-pressure technique. Demirci (2025) also prepared briquettes using cow dung, hazelnut shells and molasses and investigated the properties using response surface methodology. It was found that the quantity of hazelnut shell used had an positive effect on the heating value, molasses content improved the shatter index, whilst increasing cow dung increased the ash content. Alonge and Obayopo (2025) produced briquettes from high density wood and low density wood mixed with palm kernel shell. The briquette energy content increased as palm kernel shell content was increased.

Ajith Kumar and Ramesh (2022) studied cashew nutshell briquettes quality using binders and found that the most influencing factors were the starch content and drying duration. Ossei-Bremang et al. (2023) produced composite briquettes from different mix ratios of decanter cake and palm kernel shell. RSM was utilized to identify the physiochemical characteristics that physiochemical characteristics of composite briquette with the best calorific values. Samomsa et al. (2022) determined used simplex centroid mixture design to identify the best combination of corncobs, cassava peels, and plantain peels for composite briquettes. The quantity of cassava peels positively influenced the strength but had negative effect on the calorific value and degradation rate of the briquettes. Plantain peels slightly affected the degradation rate but had negligible effect on the strength and heating value of composite briquettes.

Adu-Poku et al. (2022) utilized a Box-Behnken RSM to study the characteristics of briquettes from sawdust, palm kernel shells, corncobs, and rice husk. Material type, particle size and pressure significantly affected relaxed density and impact resistance. Orisaleye et al. (2022a) developed predictive models and concluded that temperature, hold time and pressure significantly influenced briquette density. An optimization study was carried out on briquettes produced from forest residues by Fadele et al. (2021) using RSM. The study identified the optimum mixing ratio of jatropha seed shells with wood shavings, and the optimum binder quantity for the briquettes. Abayomi et al. (2025) utilized response RSM to optimize the production of binder-free briquettes from jujube seed shells. The study found that fine particle sizes coupled with pre-heat temperatures between 65 and 75 °C and compaction pressures up to 100 MPa yielded briquettes with optimal characteristics.

The improvement of the physico-mechanical and chemical characteristics of briquettes produced from agricultural residues is important to produce high-quality and inexpensive briquettes. Muazu and Stegemann (2015) stated that there is a need to understand the factors underlying biomass variability. Moreso, every biomass type has a set of optimum manufacturing parameters which are required for optimum briquette quality. Several studies have considered the effects of singular densification variables on the properties of briquettes without considering the effect of interacting densification variables. Some studies have considered the effect of densification variables on individual qualities of biomass briquettes but very few studies have investigated the multi-response optimization that considers simultaneously optimizing multiple qualities of the briquettes. A comprehensive study on the effect of densification variables and their interaction with the aim of determining how they simultaneously optimize various briquette properties would be beneficial for the development of appropriate briquetting technologies. This study aims to study the effects of densification variables on physical and mechanical properties of corncob briquettes using RSM, and to identify the optimum parameters for the production of high-quality briquettes.

2. Methodology

2.1 Biomass source and processing

Corncobs were used in this study for production of briquettes in a uniaxial briquetting press. In Nigeria, corncobs are available in abundance with quantity reaching up to 13.3 million tonnes per year (Ojolo and Orisaleye, 2012b). Corncobs were acquired from small-scale processing factories located in Osun State, South-West Nigeria. The collected cobs were air-dried and then milled using a biomass size reduction machine shown in Figure 1 and operated as stated by Orisaleye et al. (2022b). The milled corncobs were sieved into different particle sizes as required by the experimental designs as required for the production of briquettes.

Figure 1: Biomass size reduction machine used for milling corncobs

2.2 Description and operation of uniaxial laboratory briquetting press

The uniaxial laboratory press used in compacting corncobs into briquettes consist of a hydraulic jack, moulding chamber or die of 60 mm wrapped with a heating band, and a cover plate which are held in position by tie bars. The briquetting press is shown in Figure 2. To produce briquettes, the heating band around the die of the briquetting press was powered to heat it to the required temperature which is specified on the temperature controller. The die was opened by removing the cover plate. With the piston of the hydraulic jack positioned beneath the die, the biomass particles were loaded into the die. Thereafter, the dies were closed with the cover plate and the material within the die pressurized by operating the hydraulic jack to the required pressure which is read from the attached pressure gauge. The experimental set up was allowed to remain for a required hold time. After the period had elapsed, the cover plate of the press was removed to eject the briquette from the die through continued operation of the hydraulic jack. The ejected briquette from the die was then allowed to cool.

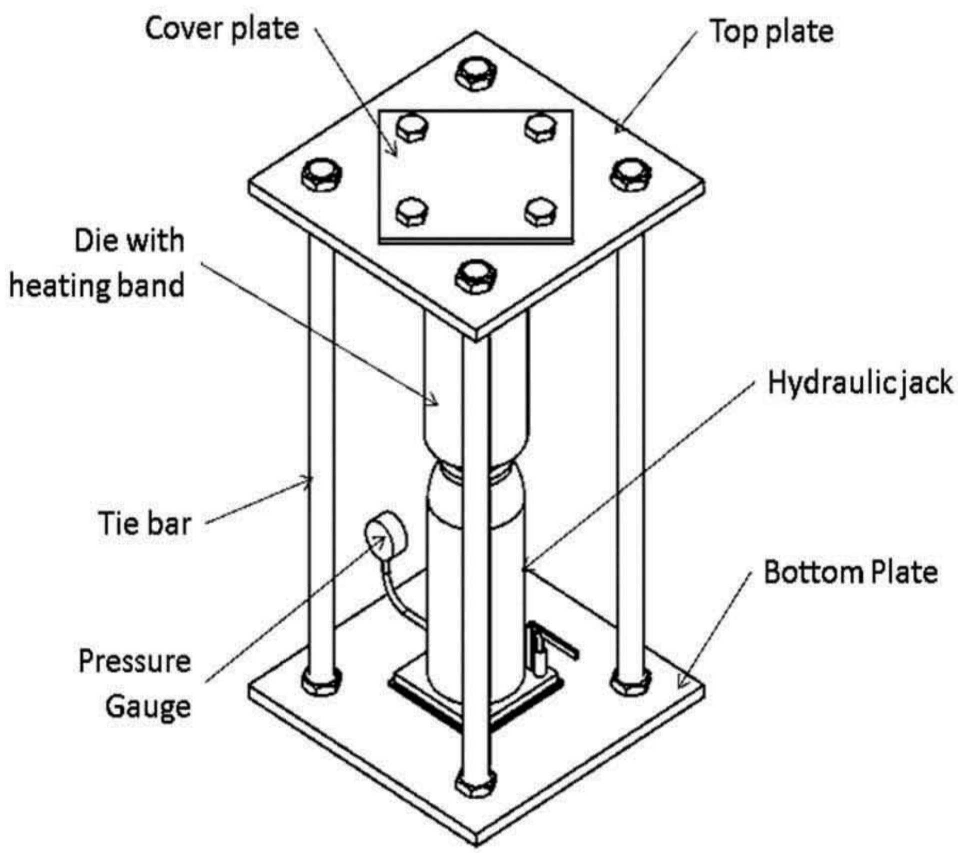


Figure 2: Laboratory scale press used for production of corncob briquettes

2.3 Experimental design

A Box-Behnken RSM experimental design was utilized to study the effects of densification variables on properties of corncob briquettes. The considered densification variables include compaction pressure, die temperature, hold time and particle size. The levels of densification variables considered are highlighted in Table 1. The Box-Behnken RSM design using the coded levels is shown in Table 2.

Table 1: Levels of variables utilized for RSM during the production of corncob and sawdust briquettes with the uniaxial press

Level	Pressure (MPa)	Temperature (°C)	Hold time (min)	Particle size (mm)
-1	9	100	7.0	< 2.26
0	12	125	11.0	2.26-3.25
1	15	150	15.0	>3.25

2.4 Quality characteristics of corncob briquettes

The physical and mechanical properties of biomass briquettes determine if briquettes are of sufficient quality. Physical properties determined include density and water resistance index (WRI). Mechanical properties include lateral compressive strength (LCS), and shatter index or impact resistance index (IRI).

2.4.1 Density of biomass briquettes

Density typifies the degree of compaction of produced briquettes. The geometry, comprising the height and diameter of briquette samples were measured using a vernier calliper. The volume of the cylindrical biomass briquette was then estimated. An electronic mass balance was utilized to measure the mass of each briquette. The briquette density was determined from the ratio of the mass to the estimated volume.

2.4.2 WRI of corncob briquettes

The WRI of the corncob briquettes was estimated by determining the difference in mass before and after briquette samples are immersed fully in water for a specified duration. The immersion of briquette samples in water was carried for 30 seconds (Birwatkar et al., 2014; Orisaleye et al., 2019). The initial and final masses of the briquette before and after full immersion in water were measured using an electronic mass balance. The resistance to water penetration in percentage (%WRI) was determined from Equation 1.

$$\%WRI = 100 - \frac{(Final\ Mass\ ) - (Initial\ mass\ )}{Initial\ mass} \qquad 1$$

2.4.3 LCS of corncob briquettes

The compressive strength was taken radially or laterally rather than longitudinally since it is a gives a more consistent reading of briquette strength according to Adeleke et al. (2021). The LCS was determined using a hydraulic compression press. The force at which the briquette fractured was determined. The lateral compressive strength was estimated using Equation 2.

$$LCS = \frac{load\ at\ fracture}{cross\text{-}sectional\ area\ of\ fracture\ plane} \qquad 2$$

2.4.4 IRI of corncob briquettes

The IRI test was carried out on briquettes produced from corncobs and sawdust briquettes. The IRI determines the durability of the briquettes when subjected to impact during handling or transportation. The test involved dropping samples of the produced briquettes from an elevation of 2 m onto a solid base. The number of drops were recorded along with the number of pieces the briquettes break into. The IRI was estimated from Equation 3 (Adeleke et al., 2021).

$$IRI = \frac{100 \times number\ of\ drops}{number\ of\ pieces} \qquad 3$$

2.5 Statistical analysis

To determine if there was a relationship between the responses, the Pearson correlation coefficient was used. Microsoft Excel was utilized to determine the Pearson correlation between responses. Design Expert version 13 was used for the RSM analysis of the Box-Behnken design. Analysis of Variance (ANOVA) was utilized in determining the significant variables to the briquette quality characteristics. Starting with full quadratic multiple regression models, response surface models were developed, and backward elimination was used to reduce the model to contain only significant parameters which do not impair the model’s ability for prediction. Surface plots were also produced to determine optimum settings for high quality biomass briquettes. Coded variables were utilized in the analysis of the RSM experimental design for corncob briquettes because of the non-numerical value of the particle sizes. The full quadratic multiple regression model has the form stated in Equation 4.

$$y = \beta_o + \sum_{i=1}^{k} \beta_i x_i + \sum_{i=1}^{k} \beta_{ii} x_i^2 + \sum_{i=1}^{k-1} \sum_{j=i+1}^{k} \beta_{ij} x_i x_j + \varepsilon$$

where β_o, β_i, β_{ii} and β_{ij} are the regression coefficients, x_i represents the model linear terms, $x_i x_j$ represents the model interaction terms, x_i^2 represents the model square terms, while ε is the error. The reliability of the response surface models in carrying out the multi-response prediction was assessed by uncertainty analysis using statistical interval estimates. This also validates the robustness of the predicted optimal conditions. The uncertainty analysis utilizes the confidence intervals (CI), prediction intervals (PI) and the tolerance intervals (TI). CI is the uncertainty around the mean responses, PI is the range within which individual future observations are expected to fall, and TI provides the range that will contain 99% of all future observations. CI, PI and TI were estimated using confidence level of 95%.

2.6 Technoeconomic analysis

A technoeconomic analysis was carried to evaluate the economic viability of producing briquettes using the uniaxial briquetting press. The approach used adopts standard methods utilized in existing studies. The mass-energy balance and cost modelling were considered for the estimation of levelized cost of briquettes. Basic assumption was that the uniaxial briquetting machine, was suitable for micro-scale production of briquettes. The process flow included the collection of the corncobs, drying, size reduction, densification in the heated piston press, cooling and bagging. Feedstock was assumed to be delivered at 25% moisture content and dried to 15% moisture content. Since it is a micro-scale production, the drying reliance was sun-drying and air-drying. To allow for the handling losses during transportation and processing, the mass yield was assumed to be 95% of the dry feed mass. The total energy required for milling corncobs (Fu et al., 2020) and the operation of the briquetting machine (Bot et al., 2022) was taken to be 1.5 kWh/kg.
Feedstocks were benchmarked at a collection rate of 50 USD per tonne which is constituted majorly by transportation and loading cost. Electricity was assumed to be supplied from the grid and at a cost of 0.15 USD/kWh. Since the equipment is suitable for micro-scale production, the labour cost was considered to be the average rural Nigerian household income of 200 USD/month (Ushie and Bassey, 2024; CBN, 2025). The capital investment was based on the cost of the development and installation of equipment approximated to be 550 USD An allowance for consumables and maintenance was 5% of the annual capital investment. Overheads were taken to be 10% of the operating expenses. An assumed weighted average cost of capital for a 10-year life cycle of the equipment was taken as 14% (Dato et al., 2025), and the capital recovery factor (Matwani et al., 2013) was used to determine the annualized capital expenses. The operating expenses were obtained from the sum of the costs of feed, power, labour, maintenance, and consumables. The levelized cost of the briquette was determined from the sum of the direct operating expenses, overheads and the capital expenses.

3. Results and Discussion

The responses from the design of experiment are presented in Table 2. The responses are density, WRI, LCS and IRI. From the analysis which follows, the effects of the densification variables on the responses were determined. From Table 2, it is seen the density of corncob briquettes ranged between 400 and 770 kg/m^3 whilst the WRI had values between 1.13 and 66.47%. The LCS was between 0.05 and 2.39 MPa and IRI had values from 5 to 200. The values obtained falls within the range of values recommended in literature. The highest density was observed with material having the highest pressure and the lowest particle size. This is reasonably expected because higher pressures force the particles closer to reduce the volume of the mass resulting in higher density. It is observed that the lower densities of the briquettes were obtained at low pressures and low to medium particle sizes. Similarly, smaller particle sizes limit the occurrence of voids within the briquettes and ensures that the particles are tightly packed to achieve higher density of briquettes. The LCS and IRI had higher values with smaller particle sizes implying that better particle-to-particle bonding occurs with the utilization of smaller particle sizes with medium to high levels of other densification variables. For the water resistance index, there is no derivable pattern of variation of the property with the densification variables from Table 2, implying that it is stochastic.

Table 2: Box-Behnken RSM experimental design and responses obtained for the quality parameters of corncob briquettes with the uniaxial laboratory briquetting press

Exp. No.	Press. (MPa)	Temp. (°C)	Hold time (min)	Particle size (mm)	Density (g/cm^3)	WRI (%)	LCS (MPa)	IRI
1	0	1	0	1	0.51	63.36	0.10	6.7
2	1	0	0	-1	0.77	29.24	2.39	200.0
3	0	0	-1	-1	0.67	42.35	1.33	150.0
4	0	1	0	-1	0.66	29.37	1.28	100.0
5	-1	0	-1	0	0.52	16.87	0.38	20.0
6	-1	0	0	1	0.44	58.33	0.05	6.7
7	1	0	-1	0	0.68	8.81	1.10	66.7
8	0	0	0	0	0.60	45.02	0.90	50.0
9	1	-1	0	0	0.55	1.13	0.70	40.0
10	0	0	0	0	0.58	34.21	0.73	50.0
11	0	1	1	0	0.57	35.7	1.28	33.3
12	0	-1	0	-1	0.59	2.46	1.18	42.9
13	1	0	1	0	0.72	47.42	1.11	66.7
14	0	-1	1	0	0.57	28.23	0.63	50.0
15	0	0	1	1	0.51	58.7	0.16	9.1
16	0	0	1	-1	0.70	25.32	2.07	150.0
17	0	0	-1	1	0.51	36.01	0.11	6.7
18	0	0	0	0	0.68	66.47	0.89	50.0
19	0	-1	-1	0	0.55	15.15	0.55	33.3
20	1	0	0	1	0.58	55.19	0.42	5.9
21	-1	0	0	-1	0.59	35.58	0.84	100.0
22	0	1	-1	0	0.46	42.77	0.64	50.0
23	-1	1	0	0	0.40	22.55	0.41	12.5
24	-1	0	1	0	0.60	62.59	0.78	40.0
25	0	-1	0	1	0.57	54.38	0.22	5.0
26	-1	-1	0	0	0.45	9.93	0.16	11.1
27	1	1	0	0	0.56	33.74	0.91	50.0

The correlation matrix of the Pearson correlation coefficients is shown in Table 3 for the comparison of the responses. It is observed from the table that the WRI has weak positive correlation with the density and weak negative correlations with the LCS and IRI. However, the LCS and IRI have strong correlations with the density. Consequently, as shown on Table 3, there is strong correlation between LCS and IRI. The strong positive correlation of the LCS with IRI is largely because the densification variables had similar effect on both the LCS and IRI. It may be implied, therefore, that either the LCS and IRI could be a measure of the mechanical integrity of the briquettes. Similarly, the density of the briquettes may also provide information on whether the briquettes have been adequately formed since it has strong positive correlation with both the LCS and IRI. Kpalo et al. (2020) also found that the density had significant positive correlations with the compressive strength. However, contrary to findings presented in Table 3, Kpalo et al. (2020) observed a significant positive correlation between density and WRI of briquettes. Spirchez et al. (2018) also observed a slight correlation between briquette density and mechanical properties. In line with observations in this study, Rejdak et al. (2019), noted that mechanical durability had strong correlation with compressive strength. Similar observations to this study on the correlations of density with compressive strength were made by Rhen et al. (2005), Gendek et al. (2018) and Taulbee et al. (2009).

Table 3: Correlation matrix of Pearson correlation coefficients for the quality characteristics of the corncob briquettes

	Density	WRI	LCS	IRI
Density	1.0000			
WRI	0.0193	1.0000		
LCS	0.8110	-0.2409	1.0000	
IRI	0.7604	-0.1643	0.8994	1.0000

3.1 Effects of variables on density of corncob briquettes

Table 4 shows the ANOVA for density of corncob briquettes where the model has been reduced by eliminating least significant higher order terms using backward elimination. From the ANOVA in Table 4, the applied pressure and particle size are statistically significant ($p < 0.05$) to corncob briquette density. The square term of the temperature is also significant ($p < 0.05$) to the density and the hold time is observed to be significant at a lower level of confidence ($p < 0.1$). It had been seen that the highest density occurred at the highest levels of pressure and particle size. The ANOVA clearly shows that the two variables are very crucial to the density and need to be appropriately set to obtain optimum density. The significance of the square term of the temperature implies that the temperature influences the density of the briquettes in a manner that is non-linear, compared to pressure and particle size that have a linear relationship with the density. The hold time is useful in allowing the relaxation of the briquettes to prevent spring-back due to the viscoelastic properties of the biomass material which could result in reduced density. From earlier studies, Aransiola et al. (2019) found that the compaction pressure was significant to the compressed density of carbonized briquettes using different binders. Jekayinfa et al. (2019) and Orisaleye et al. (2018b) found that briquette density was significantly influenced by pressure, temperature and particle size. Similar to the observations in this study, Orisaleye et al. (2022a) observed that hold time and temperature were significant terms influencing density of abura sawdust briquettes. Muazu and Stegemann (2015) identified that higher pressure had positive influence on briquette density whilst Okot et al. (2018) noted that density significantly increased with increasing compaction temperature and pressure. Orisaleye et al. (2023) also noted that temperature was one of the significant terms which determined the density of poplar briquettes.

Table 4: ANOVA for the density of corncob briquettes

Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	0.1754	7	0.0251	13.96	< 0.0001
P	0.0616	1	0.0616	34.34	< 0.0001
T	0.0012	1	0.0012	0.6686	0.4237
H	0.0065	1	0.0065	3.64	0.0716
S	0.0616	1	0.0616	34.34	< 0.0001
T×S	0.0042	1	0.0042	2.35	0.1414
P^2	0.0043	1	0.0043	2.42	0.1364
T^2	0.0395	1	0.0395	22.00	0.0002
Residual	0.0341	19	0.0018		
Lack of Fit	0.0285	17	0.0017	0.5988	0.7823
Pure Error	0.0056	2	0.0028		
Cor Total	0.2095	26			

From Table 4, the coefficient of regression, R^2, of the model is 83.72% with adjusted R^2 of 77.73% and is statistically significant (p < 0.05). The high values of the R^2 and the adjusted R^2 show that there are sufficient terms in the model, and the model is able to predict the density of the briquettes. The model for the briquette density using coded variables is stated in Equation 5.

$$\rho = 0.62 + 0.07P - 0.01T + 0.02H - 0.07S - 0.03TS - 0.03P^2 - 0.08T^2 \qquad 5$$

Using Equation 5, the predicted variables can be estimated. The plot of predicted and actual variables for each run from the experimental design is presented in Figure 3. The figure illustrates that the model is a good predictor of the density of the corncob briquettes. The surface plots presenting the effects of the variables on briquette density are presented in Figure 4. From the plots, the maximum settings of pressure and hold time, together with the minimum setting of particle size resulted in highest density values of corncob briquettes. It had been highlighted that the pressure reduces the volume of the compressed mass which results in higher density. The hold time also plays a part in increasing the briquette density as it allows the compressed mass to retain its shape and size to prevent possible spring-back of the briquettes after the pressure exerted is removed. The value of the density increases with temperature before decreasing. The decrease of the density with increase in temperature may signal the degradation of the natural binding components of the biomass briquettes. This results in insufficient particle-to-particle binding and the inability of the briquette to retain the compressed shape after the release of the pressure. Orisaleye et al. (2022a) noted that elevated levels of hold time and temperature resulted in increased density of abura sawdust briquettes but the density initially increases with pressure before decreasing. Tumuluru et al. (2015) showed that elevated die temperature and compaction pressure maximized the density of briquettes from straws of oat, canola, wheat and barley straw.

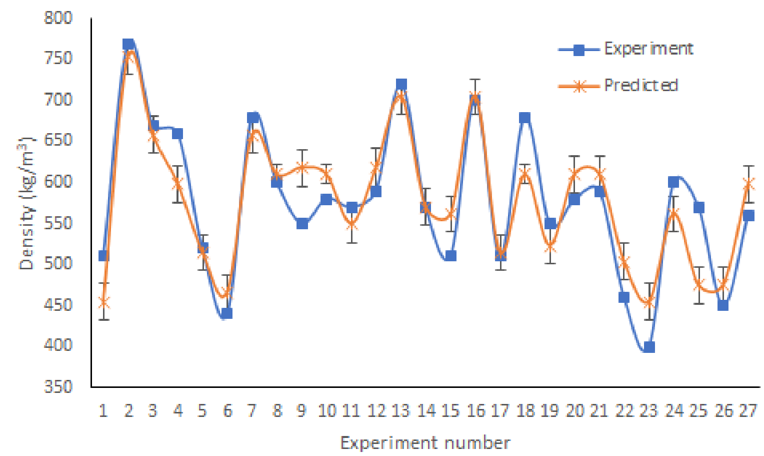
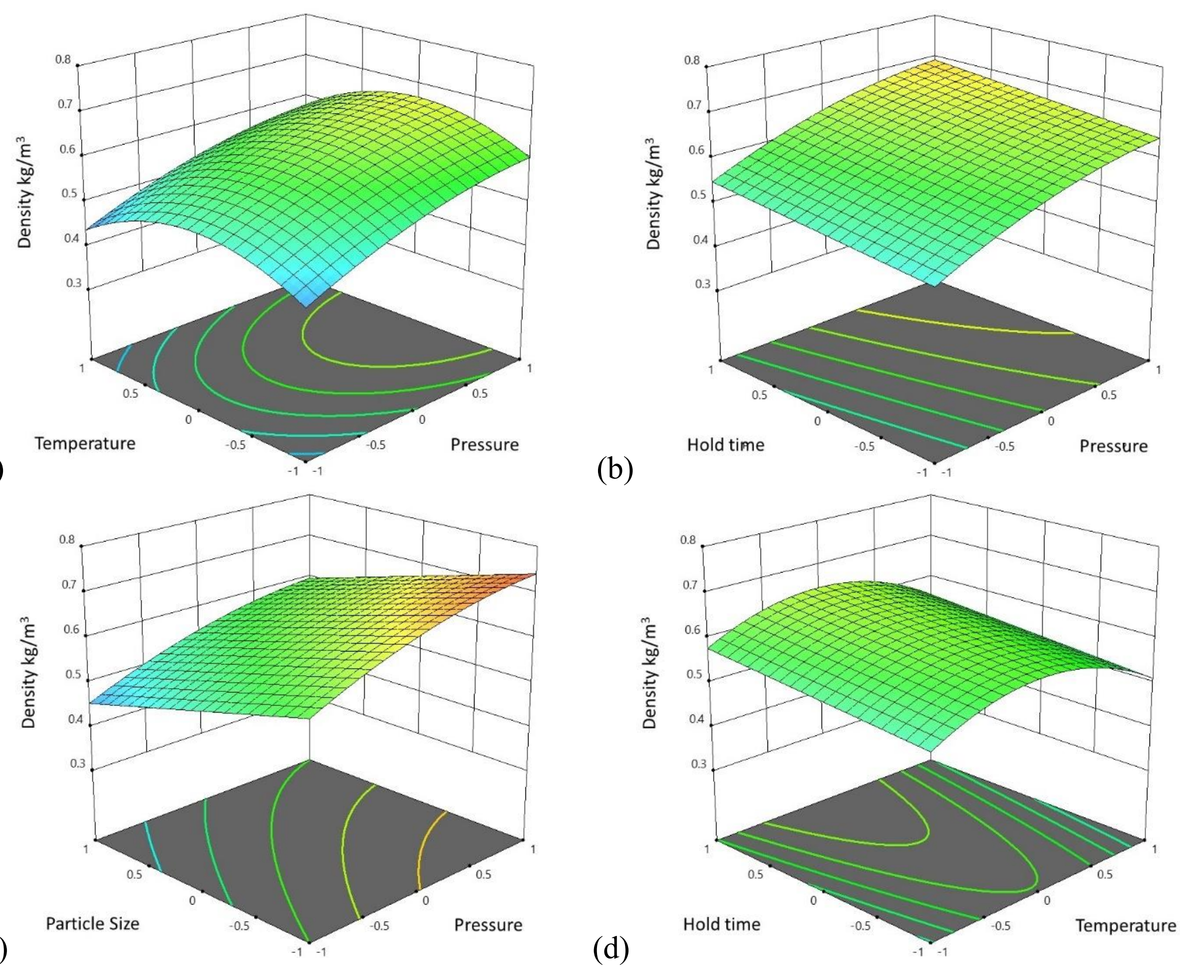
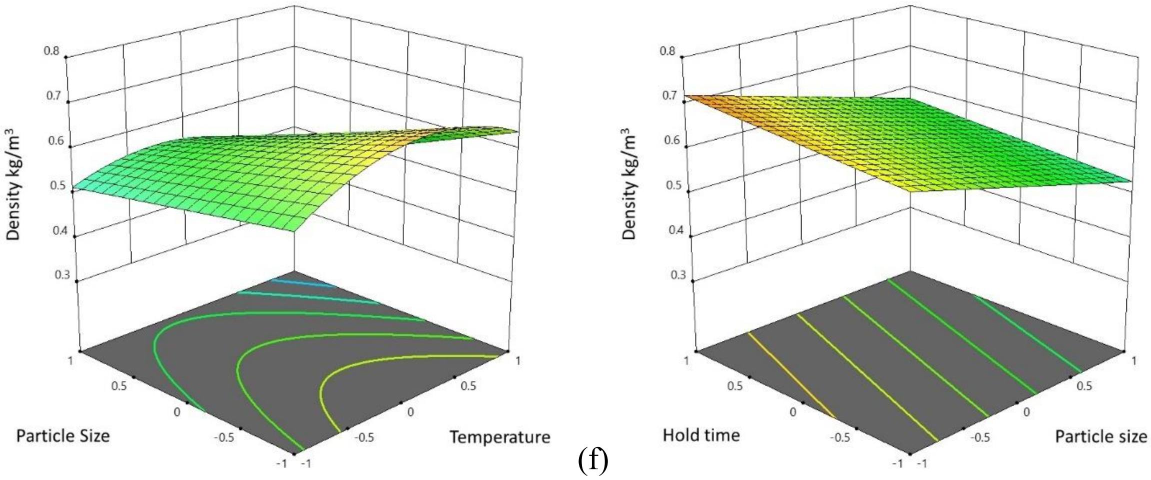


Figure 3: Comparison of predicted and actual responses for the density from the Box-Behnken experimental design





(e) (f)

Figure 4: Surface plots of density against densification variables

3.2 Effects of densification variables on WRI of corncob briquettes

The ANOVA for the WRI of corncob briquettes for a reduced response surface model using backward elimination is presented in Table 5. It is seen that the temperature, particle size and hold time are significant linear terms ($p < 0.05$) to the WRI. From Table 2, it appeared that WRI was stochastic without a defined reliance on the densification variables. However, from, Table 5, the ANOVA shows that the variables do have their effects on the WRI but the p-values show that the effects are not as pronounced compared to other properties. WRI is improved with proper binding from the natural binding constituents of the biomass which is liquefied by elevated temperatures whilst increasing the hold time allows sufficient time to coat the biomass particles for proper binding at contact interfaces. The existence of voids is detrimental to the WRI of the briquettes and this is prevented by using smaller particle size to ensure proper binding and prevent large voids. From studies involving a factorial design, Orisaleye et al. (2019) also determined that hold time along with the temperature and particle size significantly influenced the WRI. Similarly, Jekayinfa et al. (2019) found that temperature and particle size were significant determinants of the WRI of briquettes. Orisaleye et al. (2023) noted that temperature significantly affected the water resistance of poplar briquettes. Adu-Poku (2022) observed that the WRI was significantly different for different pressure and particle size but interactions did not significantly affect the WRI.

Table 5: ANOVA for the WRI of corncob briquettes

Source	Sum of Squares	Df	Mean Square	F-value	p-value
Model	6337.97	7	905.42	5.60	0.0013
P	76.61	1	76.61	0.4736	0.4997
T	1125.40	1	1125.40	6.96	0.0162
H	768.00	1	768.00	4.75	0.0421
S	2177.56	1	2177.56	13.46	0.0016
H×S	394.42	1	394.42	2.44	0.1349
P^2	626.78	1	626.78	3.87	0.0638
T^2	1483.02	1	1483.02	9.17	0.0069
Residual	3073.44	19	161.76		
Lack of Fit	2534.22	17	149.07	0.5529	0.8060

Pure Error	539.22	2	269.61
Cor Total	9411.41	26	

The response surface model, in coded variables, is presented in Equation 6. From the ANOVA presented in Table 4, the model is significant. The R^2 and adjusted R^2 are 67.34% and 55.31%, respectively signifying that the model has adequate parameters and could reasonably predict the WRI of the briquettes. Predicted values of the WRI are compared with actual experimental values in Figure 5. It is evident that the predicted and actual values follow similar trend and the model describes the effect of the variables on the WRI effectively.

$$WRI = 46.75 - 2.53P + 9.68T + 8.00H + 13.47S + 9.93HS - 9.90P^2 - 15.22T^2 \quad (6)$$

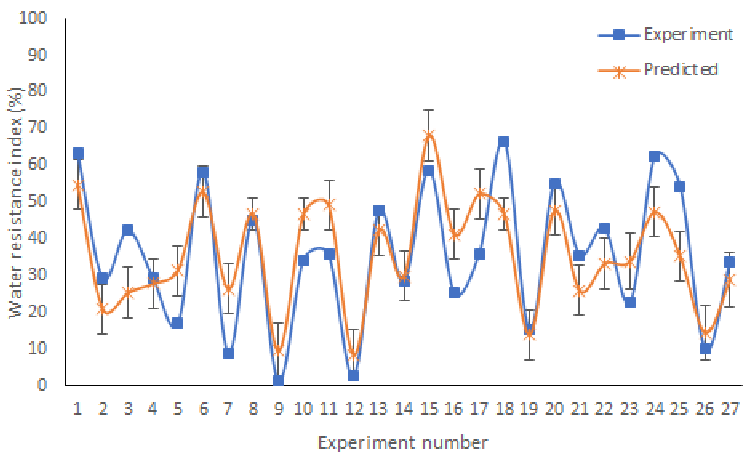


Figure 5: Comparison of predicted and actual responses for the WRI from the Box-Behnken experimental design

The response surface plots for the WRI are shown in Figure 6. From the plots, it is shown that the WRI increases with pressure and temperature before decreasing. It's also observed that the particle size has negligible effect on the WRI at the lowest hold time, and vice versa. However, greater influence on the WRI is observed as the hold time and particle sizes are higher. As previously noted, the hold time and particle size play significant roles in the particle-to-particle binding and the reduction of voids within the briquette structure which prevent the percolation of moisture in the briquettes. A previous study by Anggraeni et al. (2021) stated that briquettes produced from large particle sizes have poor water resistance. Abdulrasheed et al. (2015) also noted that the WRI of sawdust briquettes bonded with styrofoam increased with increase in pressure.

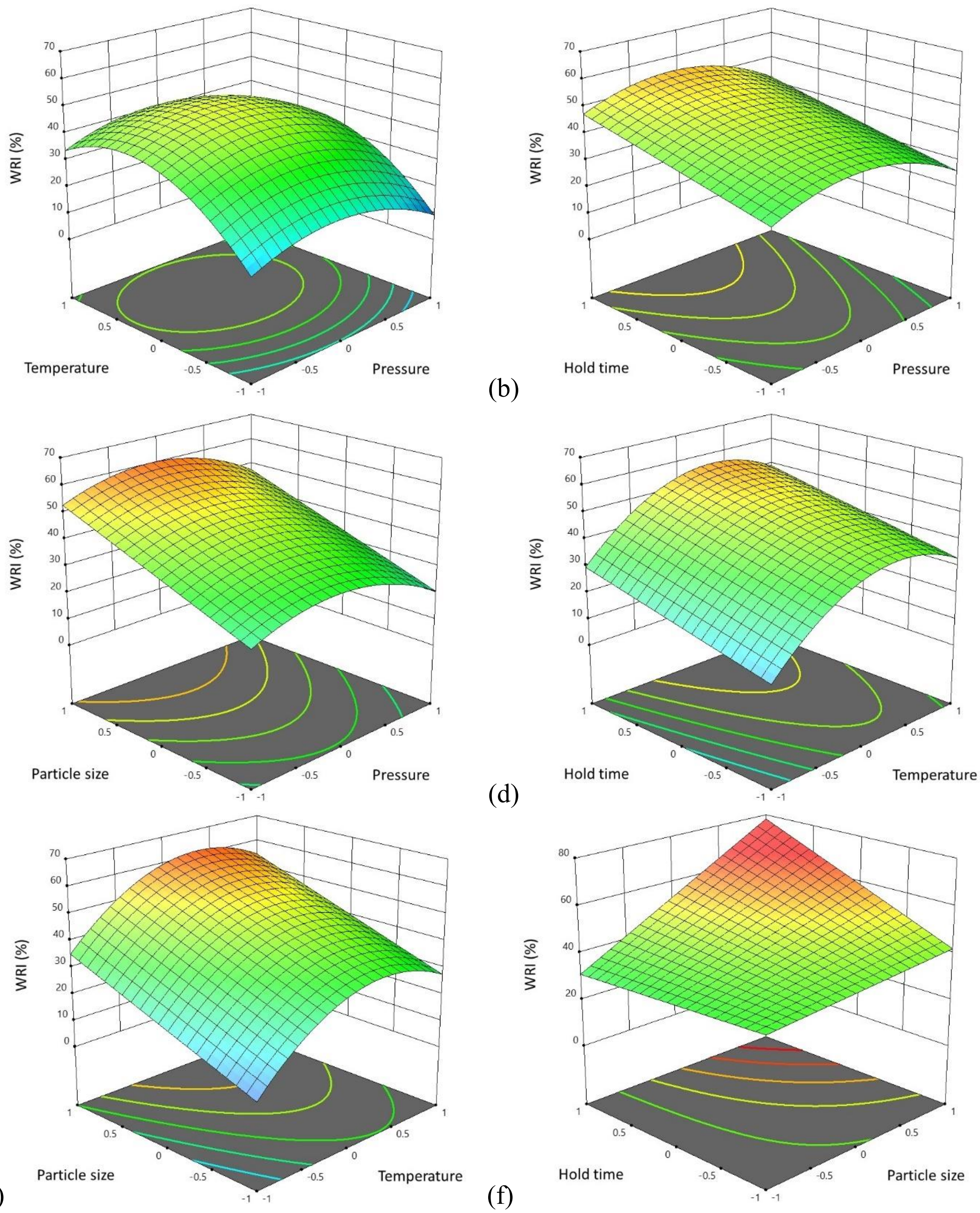


Figure 6: Surface plots of WRI against densification variables

3.3 Effects of densification variables on LCS of corncob briquettes

The ANOVA presented in Table 6 shows that the pressure, hold time and particle size are significant linear terms ($p < 0.05$) to the LCS of corncob briquettes. At a higher level of significance ($p < 0.1$), the temperature is also a significant linear term to the LCS. Significant interaction ($p < 0.05$) occurs between the pressure and particle size to determine the LCS. The LCS is a measure of how the briquettes resist crushing loads. The briquettes will need to be compact with good internal bond strength to resist external loads. To achieve this, adequate temperature ensures that the natural binding component of the biomass is liquefied. In addition, high pressure is needed to press the particles close together and press the binder out of the cells to coat the surface of the particles. The hold time allows the compacted briquette to set and retain stability under pressure. The interplay of the significant variables results in the determination of the LCS of the briquettes. From the investigation by Aransiola et al. (2019), pressure was significant for determining the compressive strength of carbonized corncob briquettes. Muazu and Stegemann (2015) found that high compaction pressure positively influenced compressive strength of rice husk and corncob briquettes. Muazu and Stegemann (2017), however, noted that the significance of utilizing higher compaction pressure on compressive strength was not apparent for briquettes produced from blends of rice husk, corncob and bagasse using binders. Zhang and Guo (2014) stated that particle size and pressure had the greatest effect on compressive strength of briquettes which is similar to findings in this study.

Table 6: ANOVA for the LCS of corncob briquettes

Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	7.98	8	0.9978	32.73	< 0.0001
P	1.34	1	1.34	43.95	< 0.0001
T	0.1160	1	0.1160	3.81	0.0668
H	0.3072	1	0.3072	10.08	0.0052
S	5.37	1	5.37	176.26	< 0.0001
P×S	0.3481	1	0.3481	11.42	0.0033
T×H	0.0784	1	0.0784	2.57	0.1262
H×S	0.1190	1	0.1190	3.90	0.0637
T^2	0.3006	1	0.3006	9.86	0.0057
Residual	0.5488	18	0.0305		
Lack of Fit	0.5306	16	0.0332	3.64	0.2365
Pure Error	0.0182	2	0.0091		
Cor Total	8.53	26			

The reduced response surface model which describes the relationship between the parameters in the ANOVA in Table 6 is presented in Equation 7. For the developed empirical model, the R^2 and adjusted R^2 are 93.57% and 90.71%, respectively. The coefficients of regression obtained show that the response surface model has adequate parameters and is suitable for the prediction of the LCS. The model is checked by comparing the predicted values of the LCS with the actual values from experimental observations. The comparison is presented in Figure 7. As observed, there is a good correlation of the predicted values from the empirical model with the experimentally observed values.

$$LCS = 0.88 + 0.33P + 0.10T + 0.16H - 0.67S - 0.30PS + 0.14TH - 0.17HS - 0.21T^2 \quad (7)$$

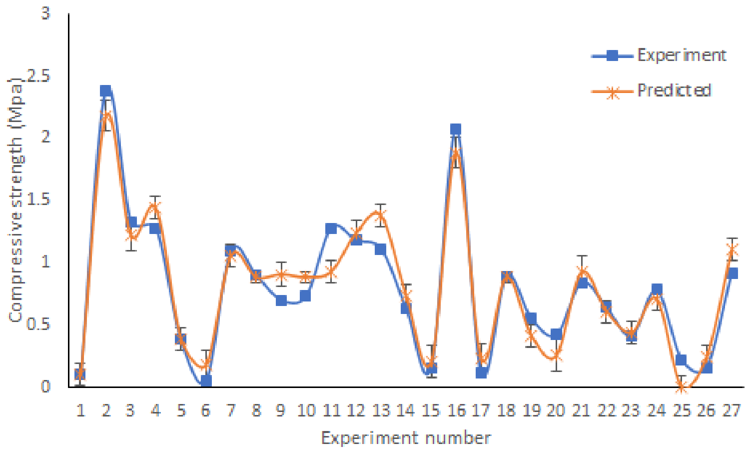
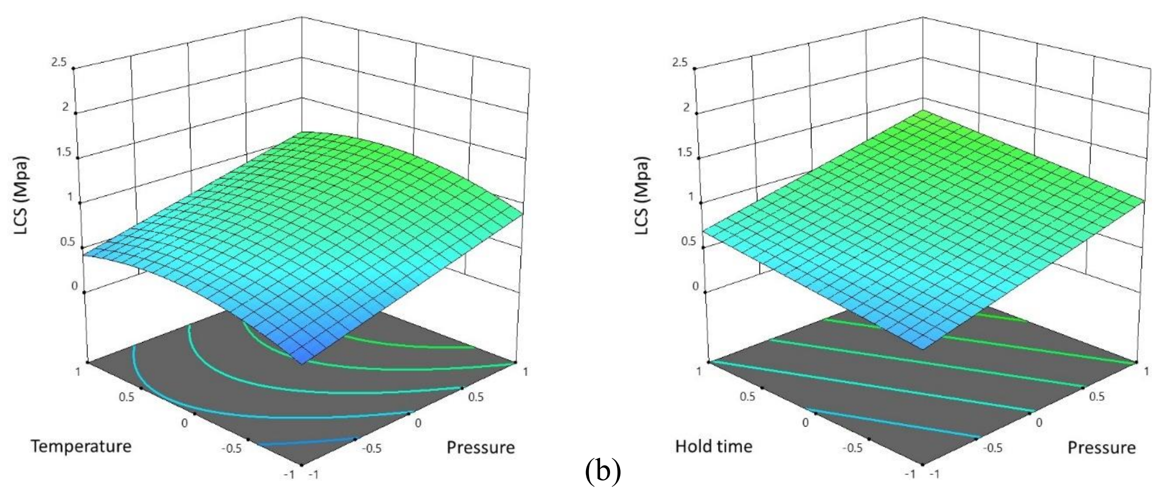


Figure 7: Comparison of predicted and actual responses for the LCS from the Box-Behnken experimental design

The relationships between the parameters in the determination of the LCS are observed from the surface plots in Figure 8. From the surface plots, higher levels of temperature, pressure and hold time resulted in higher values of LCS. However, lower levels of particle size were required for higher LCS. The variation of the LCS has been explained in terms of the roles the densification variables play in the densification process to ensure that strong briquettes capable of resisting crushing are produced. Contrary to observation in this study, Zhang and Guo (2014) observed that reducing particle size resulted in decreased compressive strength. However, in line with the observation in this study, Corpuz et al. (2017) observed that reduced particle sizes improved the compressive strength of corncob briquettes. Sam Obu et al. (2022) also made similar observation for briquettes manufactured from corncob and rice husk blend. Okot et al. (2018) noted that the compressive strength significantly increased with elevated temperature and pressure. Similarly, Ajimotokan et al. (2019), noted that briquette strength was higher with reduced particle sizes.



(a) (b)

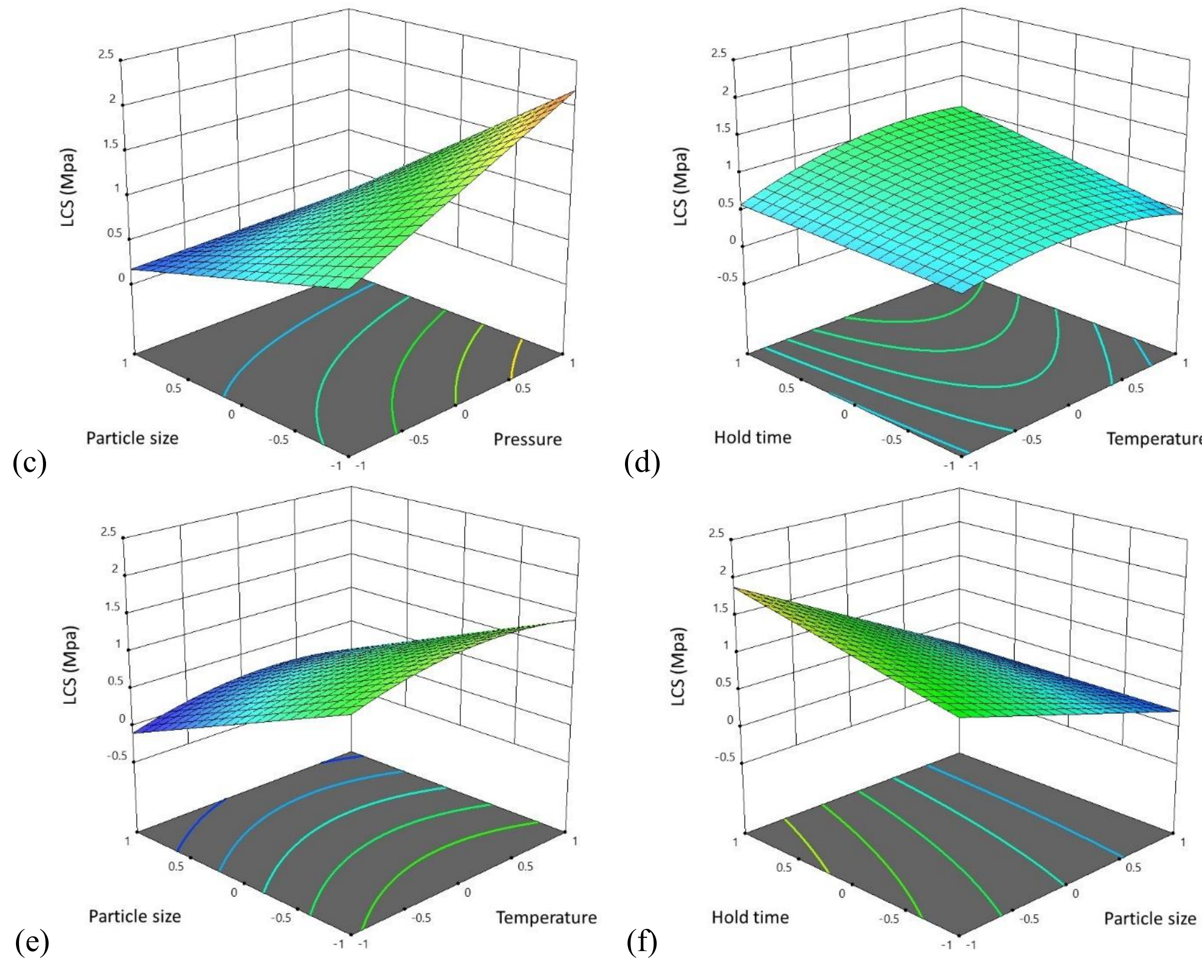


Figure 8: Surface plots of LCS against densification variables

3.4 Effects of densification variables on IRI of corncob briquettes

The IRI or drop shatter index determines the ability of briquettes to withstand shock and is also a measure of durability. Table 7 presents the ANOVA for the IRI of corncob briquettes. It is seen from the ANOVA that significant linear terms ($p < 0.05$) to the IRI are pressure and particle size. In addition to these, the interaction of pressure with particle size is significant to the IRI. From the ANOVA, it can be seen that the square terms of temperature and particle size are also statistically significant. This shows the non-linear dependence of the IRI with these variables. From the study of Zhang and Guo (2014), both pressure and particle size affected the IRI of briquettes significantly. Okot et al. (2018) showed that the temperature, pressure and particle size significantly influenced IRI of corncob briquettes. Adu-Poku et al. (2022) showed that particle size and pressure significantly determined the IRI of charred briquettes.

Table 7: ANOVA for the IRI of corncob briquettes

Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	57357.46	8	7169.68	19.93	< 0.0001
P	4760.08	1	4760.08	13.23	0.0019
T	410.67	1	410.67	1.14	0.2995
H	41.81	1	41.81	0.1162	0.7371

S	41160.65	1	41160.65	114.40	< 0.0001
P×S	2540.16	1	2540.16	7.06	0.0160
T×S	767.29	1	767.29	2.13	0.1614
T²	3939.56	1	3939.56	10.95	0.0039
S²	2241.51	1	2241.51	6.23	0.0225
Residual	6476.61	18	359.81		
Lack of Fit	6476.61	16	404.79		
Pure Error	0.0000	2	0.0000		
Cor Total	63834.07	26			

Equation 8 presents the response surface model in coded variables for the IRI of corncob briquettes. The model has values of R^2 and adjusted R^2 as 89.85% and 85.34%, respectively showing that the model is adequate to predict the IRI and there are sufficient variables within the model. Figure 9 compares the predicted and experimental values, and it is observed that predicted values are proximate to the actual values observed experimentally.

$$IRI = 54.81 + 19.92P + 5.85T + 1.87H - 58.57S - 25.20PS - 13.85TS - 24.81T^2 + 18.71S^2 \qquad 7$$

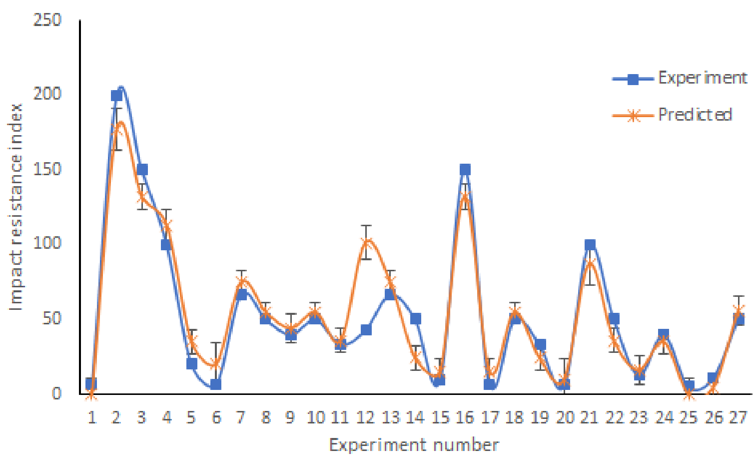


Figure 9: Comparison of predicted and actual responses for the IRI from the Box-Behnken experimental design

The response surface plots for the IRI of the corncob briquettes are shown in Figure 1-. The plots show that the high pressure and reduced particle sizes resulted in high values of IRI. The hold time did not have substantial effect on the I`RI, but IRI increased with an increase in temperature before decreasing. It is deduced that an extended hold time is not significant to increase the IRI of the briquettes produced under the conditions utilized within this study. However increased pressure and particle size are important variables that determine the IRI of the briquettes. As previously highlighted, the increasing the temperature to 150 °C could have detrimental effect on the natural binders of the biomass resulting in reduced IRI with increasing temperature beyond 125 °C. From the study of Okot et al. (2018), impact resistance increased with an increase in pressure and temperature but reduced with an increase in particle size. Zhang and Guo (2014) and Adu-Poku (2022) found that particle size reduction increased impact resistance of briquettes.

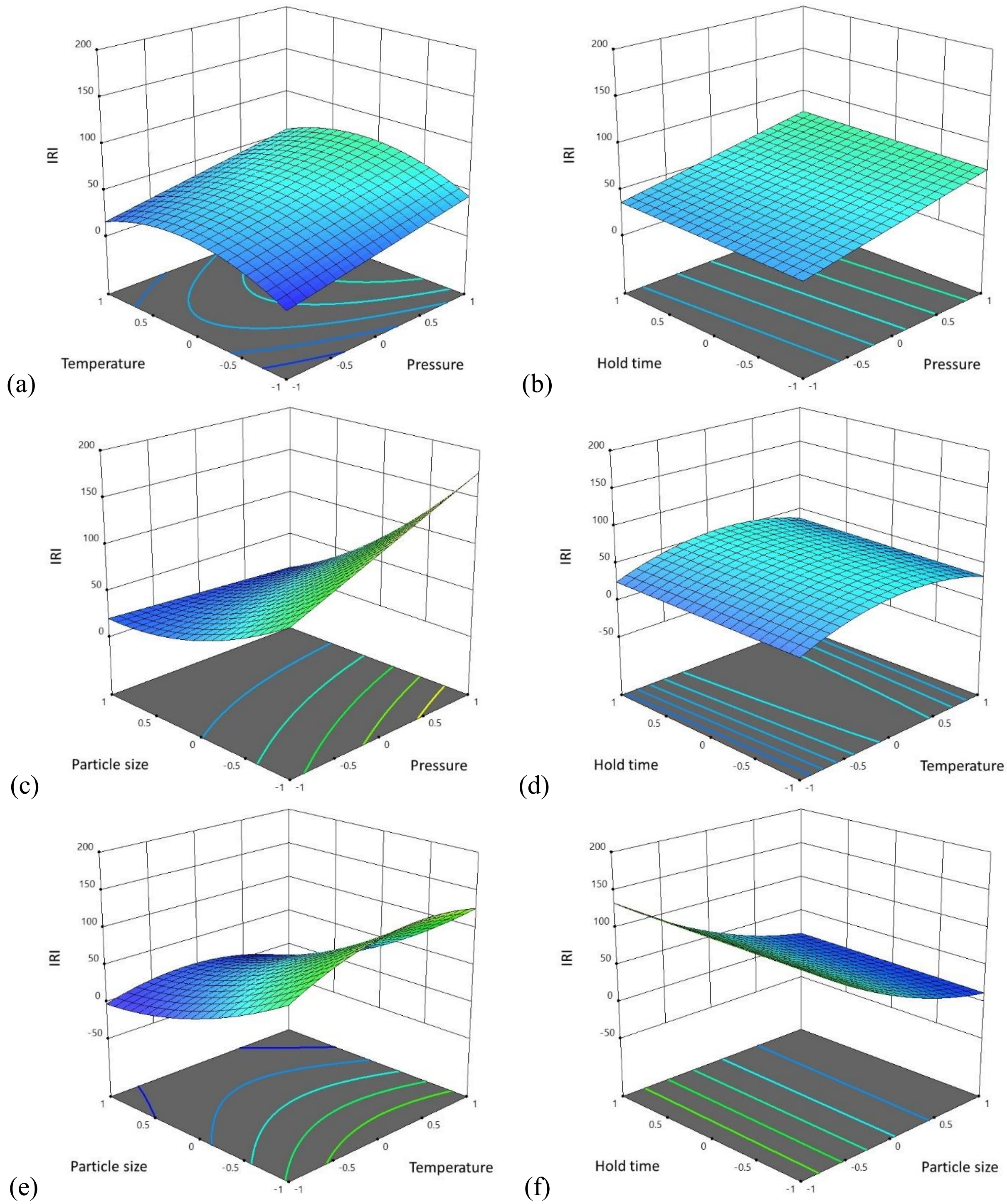


Figure 10: Surface plots of IRI against densification variables

3.5 Multi-response optimization

Multi-response optimization was carried out by maximizing the density, WRI, LCS and IRI which are desirable properties for corncob briquettes whilst the limits of the temperature, pressure, hold time and particle size were the upper and lower limits of the terms used in the Box-Behnken design. The result of the optimization is presented in Figure 12. For this solution, at the optimum setting, the density is 755.4 kg/m^3, the WRI is 26.4%, the LCS is 2.37 MPa and the IRI is 169. The optimum factors which yield this solution are pressure of 14.1 MPa, temperature of 132.9 °C, hold time of 15 minutes and particle size less than 2.36 mm. The overall desirability index of the selected solution is 0.746.

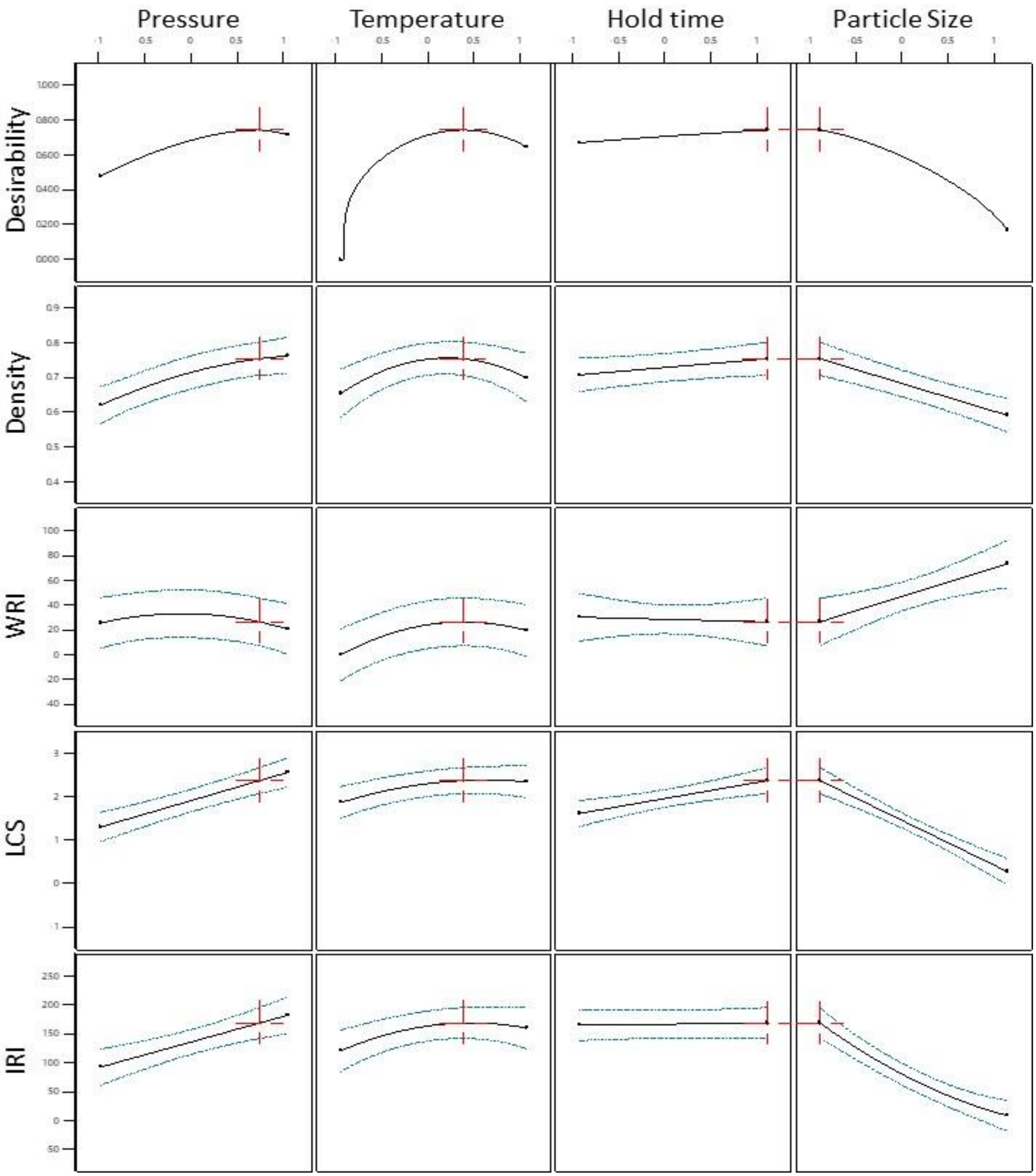


Figure 12: Optimization plots for the corncob briquettes

3.6 *Uncertainty analysis of model predictions*

The uncertainty analysis of the multi-response prediction using statistical interval estimates is presented in Table 8. The narrow 95% CI indicates that the predicted density is precise, and the model has a high level of confidence. The PI range indicates that new observations of the density of individual briquettes produced under the optimal conditions will have moderate variation. The TI shows that, under optimal conditions, 99% of briquettes are expected to have densities within acceptable range from the predicted mean density. For the WRI, there is a wide standard deviation (SD) and standard error (SE) indicated by a broad range of CI which shows that the model predicts the quality to a good degree of accuracy but with large variability. The negative range presented in Table 8 for the lower bound of the PI and TI for the WRI are unrealistic as the values cannot be negative. The negative value of the lower bounds of PI and TI further indicates the large variability of the WRI. It also suggests that the model does not properly capture new observation of the responses at the lower limits of the WRI. In this case, the lower limits of PI and TI for WRI must be adjusted such that the 95% PI low and 95% TI low are both set to zero to be physically meaningful. The LCS has narrow values for CI and PI under optimal conditions which indicate that the model predicts the quality well and there is a high possibility of repeatability of the property for new observations for individual briquettes. The TI for the LCS shows that the prediction using the response surface model captures 99% of the LCS for all briquettes. For the IRI, there is a reasonable certainty with acceptable accuracy, but high variability observed from the large values of the SD and SE. Despite the variability, the IRI lies within the expected range given by 95% PI and 95% TI. The uncertainty analysis shows the suitability of the optimized process conditions for density, LCS and IRI but also highlights that the WRI may require further quality improvement.

Table 8: Uncertainty analysis for multi-response prediction

Response	Mean	SD	SE	95% CI low	95% CI high	95% PI low	95% PI high	95% TI low	95% TI high
Density	755.4	0.042	0.023	0.707	0.803	0.654	0.856	0.566	0.945
WRI	26.4	12.72	9.17	7.19	45.58	-6.43	59.21	-34.37	87.14
LCS	2.37	0.175	0.142	2.077	2.675	1.902	2.849	1.506	3.245
IRI	168.9	18.97	12.7	142.2	195.7	120.9	217.0	79.2	258.7

3.7 *Technoeconomic analysis*

For the uniaxial press, the estimated capacity of the equipment based on the optimum hold time and allowed operating time of 5 min is estimated to be 400 kg/year considering an availability of an average of 10 hours per day. Given the assumptions made for the technoeconomic analysis, the summary of the results obtained for the production of briquettes using the uniaxial briquetting press is presented in Table 9. From the evaluation, the levelized cost of briquettes is 7.19 USD/kg if a single unit is utilized but drops to 2.79 USD/kg when 3 units of equipment are utilized. It is obvious that the uniaxial briquetting machine will be more profitable if several units are utilized. However, the sub-optimal use of equipment would cause an increase in the unit price of the briquettes. It is opined that the design of the current uniaxial briquetting machine can be modified from the current experimental configuration to allow for the production of multiple briquettes in a single run of the equipment to improve the profitability of the briquetting process. The modification of the equipment will reduce the number of equipment required for the production of higher tonnage of briquettes, thereby having an impact on the annualized capital cost and further levelling the labour cost.

Table 9: Overview of the technoeconomic evaluation for the briquetting process using the uniaxial press

Parameter	Value		
Target production (t/year)	1	0.4	1.2
Biomass supply required (t/year)	1.19	0.48	1.43
Biomass supply cost (USD/year)	59.65	23.86	71.58
Power cost (USD/year)	225.00	90.00	270.00
Labour cost (USD/year)	2400.00	2400.00	2400.00
Maintenance and consumables (USD/year)	15.82	5.27	15.82
Direct operating expenses (USD/year)	2700.47	2519.13	2757.40
Overheads (USD/year)	270.05	251.91	275.74
Capital expenses (USD/year)	316.33	105.44	316.33
Levelized cost of briquettes (USD/year)	3286.84	2876.49	3349.46
Levelized cost of briquettes (USD/kg)	3.29	7.19	2.79

4 Conclusion

The impact densification variables has on the quality characteristics of corncob briquettes were investigated in this study. The investigation utilized a Box-Behnken RSM designs. The importance of the compaction pressure, die temperature, hold time and particle size on the density, WRI, LCS and IRI were determined using ANOVA. The density of corncob briquettes ranged between 400 and 770 kg/m^3 whilst the WRI had values between 1.13 to 66.47%. The LCS had values between 0.05 and 2.39 MPa and maximum IRI values between 5 and 200. From the ANOVA, it was observed that the LCS and IRI had strong correlations with the density of the briquettes. The particle size is the single most important variable which is significant to the density, WRI, LCS and IRI. The pressure was also significant to the density and the LCS whilst the hold time was significant to the WRI and LCS. The interaction between the pressure and particle size was significant to the LCS and IRI. From surface plots, it was shown that elevated levels of pressure and hold time, together with the lowest setting of particle size resulted in highest density values of corncob briquettes. Higher levels of hold time and particle time resulted in higher WRI. Higher levels of pressure, temperature and hold time were required for higher values of LCS. Elevated levels of pressure and lower settings particle size resulted in higher values of IRI. From mutli-response optimization, the optimum density, WRI, LCS and IRI are 755.4 kg/m^3, 26.4%, 2.37 MPa and 169, respectively. The factors which yield these optimum characteristics are pressure of 14.1 MPa, temperature of 132.9 °C, hold time of 15 minutes and particle size less than 2.36 mm. The uncertainty analysis carried out by considering the CI, PI and TI shows the suitability of the optimized process conditions for density, LCS and IRI but also highlights that the WRI may require further quality improvement. The technoeconomic analysis of the briquette manufacturing by micro-scale enterprises using the uniaxial briquetting press suggests that the process could be more profitable for with the modification of the machine for higher production capacity.

Data Availability: All data generated or analysed during this study are included in this published article.

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