

Optimization of Densification Parameters for Lean Grade Subbituminous Coal

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Abstract

The densification parameters for sub-bituminous coal briquette were optimized in this study to obtain maximum drop to fracture (DF), water resistance index (WRI), and cold crushing strength (CCS). Full factorial experimental design was used to optimize the compaction pressure (14 and 56 MPa), particle size (0.5 - 1 mm), and external moisture content (3 - 10%). Briquettes were produced with a pitch binder and cured at 300°C for 60 minutes in an inert environment of a tubular furnace. The DF, WRI and CCS of the coal briquettes were carried out and mathematical models were developed between the dependent and independent variables. The results showed that particle size has the largest influence on the physicochemical properties, whereas the pressure and moisture content have minimal impact. Lower particle size implied better physicochemical properties for the coal briquettes. The optimum process parameters for maximum physicochemical properties were pressure of 56 MPa, particle size of 0.5 mm, and moisture content of 10%. The maximum DF, WRI, and CCS obtained for the coal briquettes were 170 times/2m, 97.78%, and 7.94 MPa, respectively. The mathematical models developed were validated and found useful at 95% confidence level. Coal briquettes useful for energy generation was successfully developed.

Keywords: Optimization; Particle size; Cold crushing strength; Water resistance index; Drop to fracture.

1. Introduction

Coal is one of the solid minerals that are adequately available in Nigeria [1-4]. For most metallurgical applications, coking coals are more desirable [5-7]. However, because they are very limited all over the world [8], non-coking coals are often used alternatively for several applications. Nigerian coals are majorly sub-bituminous coal, which is of lean grade [9-11]. Meanwhile, the mining of these coal grades generates a lot of fines. These huge fines are often considered as wastes, whereas they can be utilized by processing them into more useful thermochemical products [12-14]. Just as agro-waste combinations have been used in many ways [15-25], these wastes have been previously utilized in various ways. Coal fines were used as a partial replacement for cement in concrete production [26-28]. Several authors have used the combination of coal fines and agro-waste in the production of briquettes [9, 29-31]. Niu *et al.* [32] examined the possibility of generating energy from low-rank coal through low-temperature pyrolysis and discovered that the floatability and hydrophobicity of sub-bituminous coal can be enhanced by pyrolysis. Liu *et al.* [33] also produced high strength clean briquettes from coal fines through the use of polyvinyl alcohol and coal slime as binders. Predominantly, coal fines can be used to produce briquette, which can serve as replacements for coal in rotary kiln [12, 34]. In producing briquettes, several parameters such as type of binders, external moisture addition,

curing temperature, time of curing, particle sizes of the fines, and pressure are put into consideration [35-41]. These factors affect the thermomechanical properties of the briquette. In essence, if the produced briquette is not well compacted, it becomes a problem in use irrespective of its combustion properties [30,42]. Several authors have produced briquettes in numerous ways. Some produced coal briquettes with raw and treated biomass [42-44]. Briquettes have also produced from only coal fines to form coke for metallurgical applications [45]. The parameters used to produce these briquettes were selected based on the usual one factor at a time techniques and they were regarded fit as feedstock for rotary kiln and energy generation. Ajimotokan *et al.* [17] investigated the physico-mechanical properties of corncob and rice composite briquette by varying the particle sizes, mix ratios, and pressure of compaction with the usual one factor at time (OFAT) technique. The study concluded that the physico-mechanical properties of briquettes depend on the variation of the densification parameters. However, to have a comprehensive perception of the impact of these densification parameters on the physico-mechanical properties of coal briquette, a parametric study using optimization technique is thus important. Therefore, the present study is focused on the optimization of external moisture content, particle size, and pressure as densification parameters for the production of briquette from lean grade sub-bituminous coal of Nigeria origin.

2. Material and methods

2.1. Material preparation

The raw materials used in this study were lean grade sub-bituminous coal fines obtained from Okaba mine, Nigeria, and coal tar pitch binder obtained from National Metallurgical Laboratory, Jharkhand, India. The fines were oven-dried at 105°C to remove unbounded moisture. It was then screened to various particle sizes (0.5 - 1 mm). The characteristics of the Okaba coal is shown in Table 1.

Table 1. Characteristics of Okaba coal

Proximate analyses and HHV				
MC (%)	VM (%)	AC (%)	FC (%)	HHV(MJ/kg)
1.37	13.71	18.00	64.92	24.20
Ultimate analyses				
C (%)	H (%)	N (%)	S (%)	O (%)
71.47	2.88	0.90	0.71	24.04

*OC-Okaba coal, Moisture content, VM-Volatile matter, AC-Ash content, FC-Fixed carbon content, C-Carbon, H-Hydrogen, N-Nitrogen, S-Sulphur, O-Oxygen, HHV-Higher heating value

2.2. Design of experiment

The experimental design was carried out using full factorial design in Minitab 17 software [46]. Each densification was blocked with two replicates, as shown in Table 2.

Table 2. Experimental design matrix (full factorial) using MINITAB 17

Run order	Sample ID	Pres. (MPa)	Par. Size (mm)	Mois. Cont. (%)
1	14-1-3	14	1.0	3
2	14-1-10	14	1.0	10
3	14-1-3	14	1.0	3
4	56-0.5-10	56	0.5	10
5	56-0.5-10	56	0.5	10
6	14-0.5-10	14	0.5	10
7	14-0.5-10	14	0.5	10
8	56-1-10	56	1.0	10
9	14-0.5-3	14	0.5	3
10	56-1-10	56	1.0	10
11	56-0.5-3	56	0.5	3
12	14-0.5-3	14	0.5	3

Run order	Sample ID	Pres. (MPa)	Par. Size (mm)	Mois. Cont. (%)
13	56-1-3	56	1.0	3
14	14-1-10	14	1.0	10
15	56-0.5-3	56	0.5	3
16	56-1-3	56	1.0	3

The dependent variables were drop to fracture (DF), water resistance index (WRI), and cold crushing strength (CCS); while pressure (pres.), particle size (par. size), and external moisture content (mois. cont.) were the independent variables. The high and low values that were set for pressure, particle size, and moisture content were 14 and 56 MPa, 0.5 and 1.0 mm, and 3 and 10%, respectively. Sixteen densification runs were carried out and each was treated as separate densification.

2.3. Densification process

The coal fines (25 g), pitch (10% of coal weight), and water (varied) were thoroughly mixed to obtain homogeneity. The mixture was poured into a 25 mm diameter cylindrical steel die and compressed with experimental designed pressure (varied). The sample was ejected from the mold and cured room temperature. It was further cured in an inert environment at 300°C for 60 minutes in a tabular furnace.

2.4. Analyses of the physicochemical properties

2.4.1. Drop to fracture (DF)

This was obtained by recording the number of times the briquette was dropped from a height of 2 m before it fractured into pieces. This is an indication of the impact strength of the briquette, which is highly significant during transportation and usage in thermal plants [9,47].

2.4.2. Water resistance

The immersion of a single briquette in water was adopted to measure its resistance against water resistance according to the modified Richard's method [9,47]. A briquette of known weight (W_1) was immersed in 220 mL of distilled water in a glass that was at $30 \pm 2^\circ\text{C}$ for 30 minutes. It was then removed, cleaned, and weighed again (W_2). The change in the weight of the briquette was determined as water absorbed using Equation 1. The water resistance index (WRI) was calculated according to Equation 2.

$$\text{Water absorbed} = \frac{W_2 - W_1}{W_1} \times 100\% \quad (1)$$

$$\text{WRI}(\%) = 100 - \% \text{water absorbed} \quad (2)$$

2.4.3. Cold crushing strength

Cold crushing strength (CCS) is also known as the compressive strength of the briquette. It is the maximum compressive load applied per unit area on the briquette before it fails. The procedure for obtaining the CCS involves the use of a 10 kN Universal Testing Machine (UTM) available at National Metallurgical Laboratory, Jamshedpur, India. The procedure for testing is as reported by [9].

2.5. Statistical analyses

Semi-empirical linear models were developed between the dependent variables (pressure, particle size, and moisture contents) and independent variables (Drop to fracture, water-resistance index, and cold crushing strength) in uncoded formats and were validated with experimentation using the single-point solution from the response optimizer of Minitab 17 version.

3. Results and discussion

3.1. Physicomechanical properties

Fig. 1 displays the physicomechanical properties of the produced briquettes. It can be observed that as the pressure increased from 14 to 56 MPa, DF increased from 162.5 to 175.5 times/2m, WRI reduced from 98 to 97.66% ,while CCS increased from 7.51 to 7.52 MPa when particle size (0.5 mm) and moisture content (3%) were kept constant. Similarly, at constant pressure (14 MPa) and moisture content (3%), the DF decreased from 162.5 times/2m when the particle size was 0.5 mm to 71 times/2m when the particle size was 1 mm. Under the same condition, WRI decreased from 98% to 91.94% while CCS decreased from 7.5 to 4.48 MPa. Variation of moisture content from 0.3 to 10% at constant pressure (14 MPa) and particle size (0.5 mm) showed that the DF decreased from 162.5 to 159 times/2m.

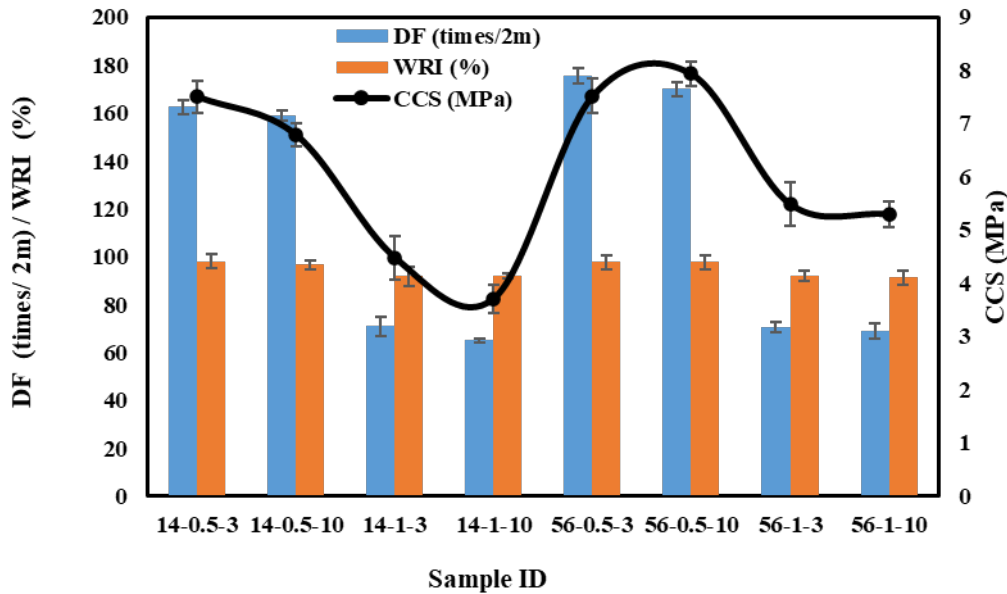
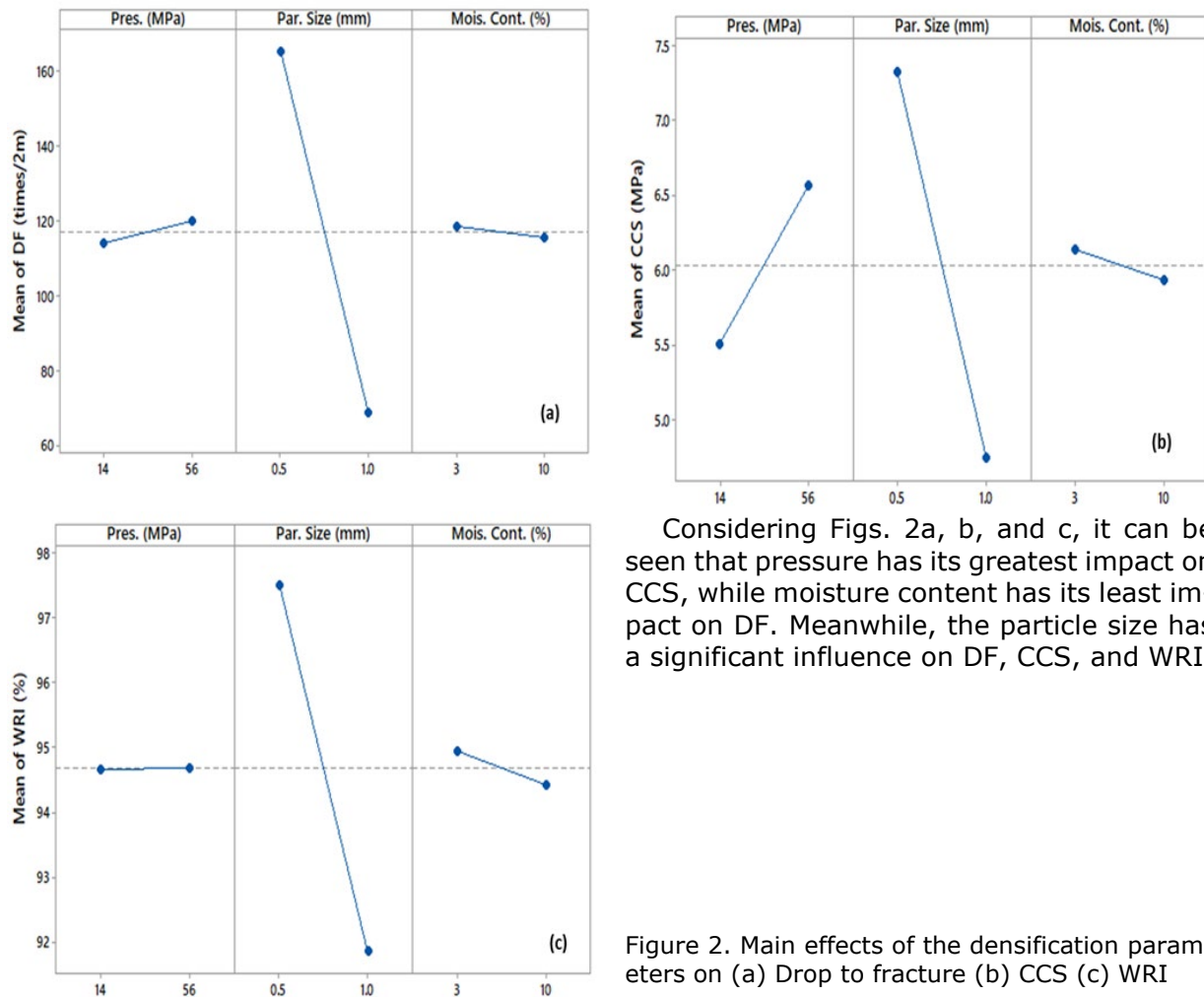


Figure 1. Physicomechanical properties of the subbituminous briquettes

The CCS of the briquette also reduced from 7.51 to 6.79 MPa while WRI reduced from 98 to 96.7%. From the results, it can be observed particle size has the largest effect on the physicomechanical properties compared to the abysmal effect of pressure and moisture content. This shows that as the particle size reduces, the DF, CCS, and WRI increased due to the reduction of the interstitial spaces between particles, thereby increasing the inter-particle bonding, which becomes more difficult to break mechanically leading to increased strength. This can be seen in Fig. 2, which shows the main effect of densification parameters on DF, CCS, and WRI. Figure 2a revealed that there is a minimal variation of DF based on pressure and moisture content, but a wide displacement about the mean was observed for particle size. Higher DFs were recorded at very low particle sizes. Densification is enhanced by a reduction in particle size and the strength of the briquette increased when it was dropped or thrown from a height. This is particularly important during transportation and loading of briquettes into combustion units of boilers and other thermal utilities that involve the briquettes been thrown from several meters, which can cause some to be crushed to pieces and not able to serve its essence effectively. Therefore, the ability of the briquette to withstand such stress is tantamount to its relevance in various applications [9,33,48]. Fig. 2b shows the response of CCS to variation in densification parameters. It shows that the pressure of compaction influenced the cold crushing strength of the briquettes. The positive slope of the graph shows that CCS increases with an increase in pressure of compaction. The higher the pressure of compaction, the smaller the inter-particle spacing and the greater the inter-particle bonding, thereby leading to more strength [43,49-51]. Gaballah *et al.* [48] attributed the increase in CCS to increase in

Vander Waals forces within the briquettes. Despite this, particle size had the greatest influence on CCS of the briquettes. The negative slope of the graph indicates that increase in particles size leads to a decrease in CCS. This again signifies that inter-particle bonding is stronger at smaller particle sizes. Conversely, there was no significant spread about the mean value of CCS based on moisture content. In the process of densification, most of the moisture content of the briquette would have been lost since the impact is not pronounced. The CCS plays an important role in the application of briquette as they are transported and stored in stacked manner whereby those at the top mount pressure on those at lower levels. Even in use, briquettes are usually placed one on another in the combustion chambers of boilers, if the CCS is low, the briquette in the lower layers may be crushed before they are eventually put in use due to compressive stress. The results of the study are in line with the findings of Czechowski *et al.* [52] who theorized that particle parking has a major influence on CCS whereas, the impact of pressure (load per unit area) was considered minor. In Fig. 2c, the response of WRI to densification parameters is visualized. The compaction pressure has no impact on WRI, while moisture content has very little impact on WRI, but again the particle size plays a key role in the WRI of coal briquettes. As the particle size gets smaller, the water retention capacity increases due to the spaces between particles that are closed up, thereby limiting pores for water to percolate [9,53].



Considering Figs. 2a, b, and c, it can be seen that pressure has its greatest impact on CCS, while moisture content has its least impact on DF. Meanwhile, the particle size has a significant influence on DF, CCS, and WRI.

Figure 2. Main effects of the densification parameters on (a) Drop to fracture (b) CCS (c) WRI

3.2. Interaction effect of densification parameters on DF, WRI, CCS

Fig. 3 (a-c) shows the surface plots of the interaction between particle size and pressure; and their implications on the physico-mechanical properties on the coal briquettes. From the

surface plots, the values of particle size and pressure needed to obtain optimum drop to fracture (Fig. 3a), cold crushing strength (Fig. 3b) and water resistance index (Fig. 3c) can be determined. It can be observed that pressure of 56 MPa and particle size of 0.5 mm are needed to obtain maximum DF, CCS, and WRI.

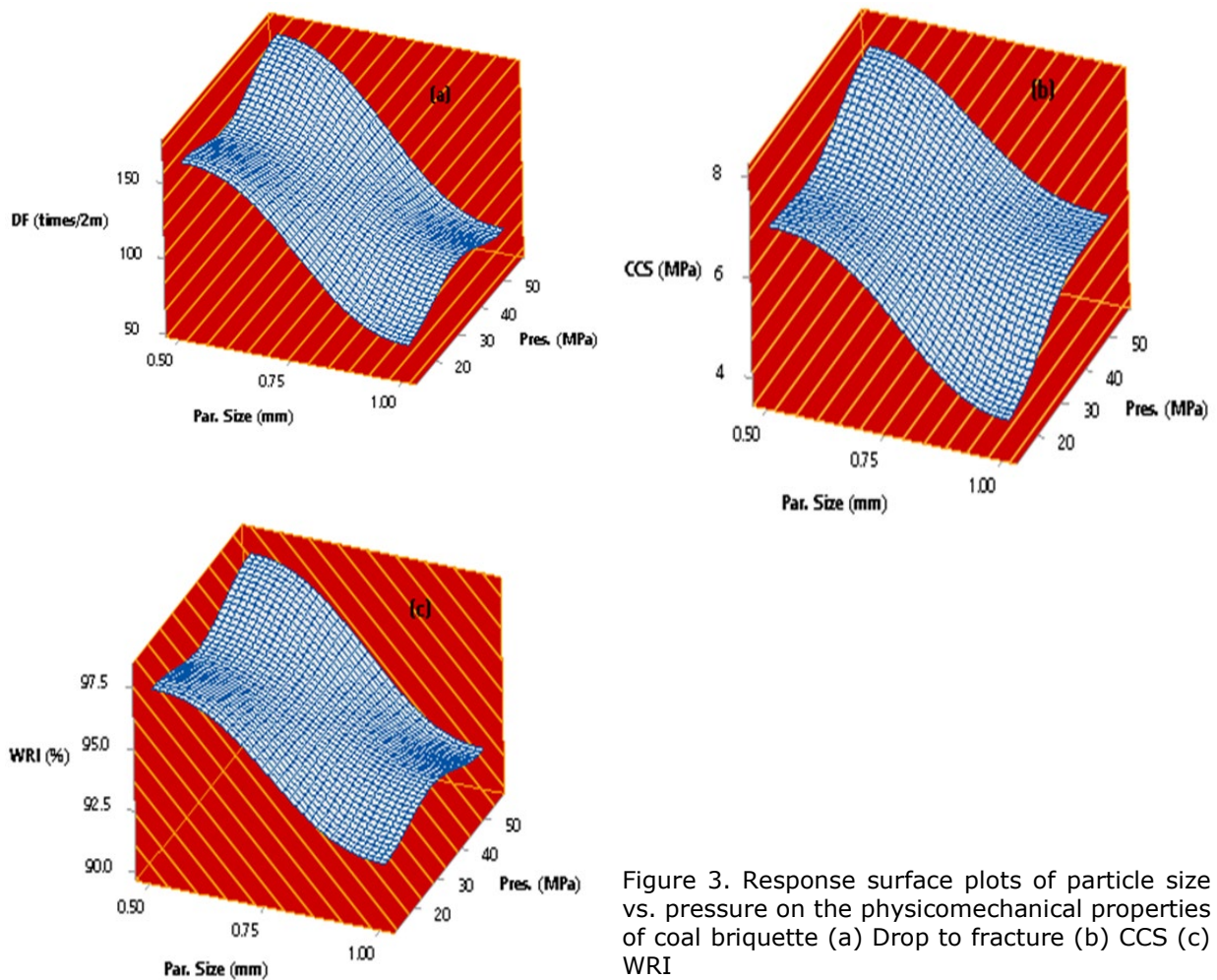


Figure 3. Response surface plots of particle size vs. pressure on the physico-mechanical properties of coal briquette (a) Drop to fracture (b) CCS (c) WRI

The interaction between particle size and moisture content is shown in Fig. 4.

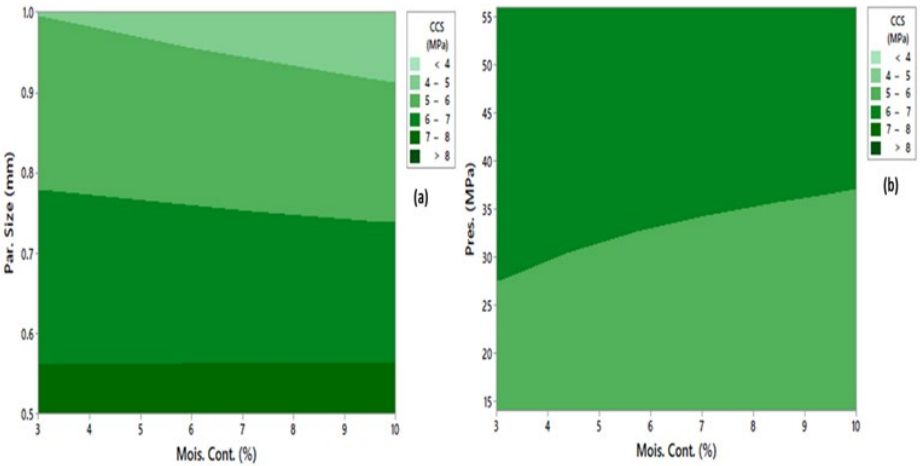


Figure 4. Interaction effects of factors on CCS (a) particle size and moisture (b) pressure and moisture contents

Figure 4a revealed that the moisture content had no significant effect as changes could only be seen as the particle size changes. Similarly, the contour interaction effect plots of pressure and moisture content in Fig. 4b shows that the moisture content has little impact on the CCS, which was used as representative of the responses. The highest strengths were recorded when the moisture content was 10%, which showed that moisture content has minimal impact on the physico-mechanical properties of briquette. High moisture content enables even mixing of the coal fines and the binder, which on compaction leads to improved strength [33].

3.3. Statistical analysis

The ANOVA results for the models are displayed in Table 3.

Table 3. Analysis of variance (ANOVA) of the models for DF, WRI and CCS of the coal briquette

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Drop to fracture: Correlation Factor (R ²): 99.88%					
Model	7	37582.9	37582.9	987.40	0.000
Linear	3	37505.2	37505.2	2299.17	0.000
Pres.	1	126.6	126.6	23.28	0.001
Par. Size	1	37345.6	37345.6	6868.15	0.000
Mois. Cont.	1	33.1	33.1	6.08	0.039
Pres.*Par. Size	1	60.1	60.1	11.05	0.010
Pres.*Mois. Cont.	1	14.1	14.1	2.59	0.146
Par. Size* Mois. Cont.	1	3.1	3.1	0.56	0.474
Pres.*Par. Size*Mois. Cont.	1	0.6	0.6	0.10	0.756
Error	8	43.6	5.4		
Total	15	37626.4			
WRI: Correlation Factor (R ²): 97.83%					
Model	7	131.066	18.724	51.53	0.000
Linear	3	128.466	42.822	117.84	0.000
Pres.	1	0.002	0.002	0.01	0.942
Par. Size	1	127.464	127.464	350.77	0.000
Mois. Cont.	1	1.000	1.000	2.75	0.136
Pres.*Par. Size	1	0.664	0.664	1.83	0.213
Pres.*Mois. Cont.	1	0.051	0.051	0.14	0.719
Par. Size* Mois. Cont.	1	0.102	0.012	0.28	0.610
Pres.*Par. Size*Mois. Cont.	1	1.782	1.782	4.90	0.058
Error	8	2.907	0.36		
Total	15	133.973			
CCS: Correlation Factor (R ²): 98.93%					
Model	7	32.1555	4.936	105.33	0.000
Linear	3	31.2072	10.4024	238.52	0.000
Pres.	1	4.4732	4.4024	102.57	0.000
Par. Size	1	26.5740	26.5740	609.32	0.000
Mois. Cont.	1	0.1600	0.1600	3.67	0.092
Pres.*Par. Size	1	0.2352	0.2352	5.39	0.049
Pres.*Mois. Cont.	1	0.3969	0.3969	9.10	0.017
Par. Size* Mois. Cont.	1	0.3136	0.3136	7.19	0.028
Pres.*Par. Size*Mois. Cont.	1	0.0025	0.0025	0.06	0.817
Error	8	0.3489	0.0436		
Total	15	32.5044			

It is evident from the results that pressure ($p = 0.001$), particle size ($p = 0.000$), moisture content ($p = 0.0390$), and a combination of pressure and particle size ($p = 0.010$) have significant influence on drop to fracture (DF). Pressure and moisture content have no significant influence on WRI. However, only particle size ($p = 0.000$) has a significant influence on WRI. Meanwhile, pressure ($p = 0.000$), particle size ($p = 0.000$), and their interaction have significant influence on the cold crushing strength.

This is confirmed by Ajimotokan *et al.* [17] that reported that the cold crushing strength of briquettes depends on the particle size and pressure of compaction. The ANOVA results further confirmed that particle size has the most significant influence on the physicochemical properties of coal briquette. Based on the ANOVA results, mathematical models for DF, WRI, and CCS are shown in Equations 3-5, respectively. The correlation factors (R^2) for all three properties (DF, WRI, and CCS) were 99.88, 97.83, and 98.93%, respectively. This shows that there is an adequate correlation of the model with the experimental data [54-57]. With these mathematical models, the DF, WRI, and CCS can be predicted given the densification parameters before experimentation. This will serve as a check to experimental results. The optimum densification parameters and responses for the briquettes, as obtained from the response optimizer of Minitab 17 is shown in Table 4.

Table 4. Response optimizer in MINITAB 17 vs. confirmatory experimental results

Densification parameters			Responses		
Pressure (MPa)	Par. size (mm)	Mois. cont. (%)	DF (times/2m)	WRI (%)	CCS (MPa)
56 ^a	0.5	10	170	97.78	7.94
56 ^b	0.5	10	168	96.60	7.66

^aPrediction obtained using response optimizer in MINITAB 17, ^bExperimental

A densification experiment was carried out using these densification parameters and the results obtained are also compared with the predicted in Table 3. The results revealed that to obtain optimum values of DF, WRI, and CCS, a pressure of 56 MPa, 10% moisture content, and 0.5 mm particle size are needed. The results further displayed that the maximum values of physicochemical properties obtained by prediction are very close to those obtained from the confirmatory densification, thereby, validating the mathematical models. The method of optimization in this study present a similar trend of results when [57] used design expert optimizer to optimize the torrefaction conditions of high energy biofuel made from oil palm biomass. Generally, the results showed that particle size has the most significant influence on the densification of sub-bituminous coal. The model developed can be used to predict the drop to fracture, cold crushing strength, and water resistance of coal briquettes.

3.4. Linear regression equations

$$DF \left(\frac{\text{times}}{2m} \right) = 117.19 - 2.81 * \text{Pres.} + 48.31 * \text{Par. Size} + 1.44 * \text{Mois. Cont.} - 1.94 \text{ Pres.* Par. Size} \quad (3)$$

$$WRI (\%) = 94.68 - 0.01 * \text{Pres.} + 2.82 * \text{Par. Size} - 0.25 * \text{Mois. Cont.} + 0.33 * \text{Pres.* Par. Size} * \text{Mois. Cont.} \quad (4)$$

$$CCS (\text{MPa}) = 6.01 + 0.53 \text{ Pres.} + 1.29 \text{ Par. Size} - 0.1 * \text{Mois. Cont.} + 0.12 \text{ Pres} * \text{Par. Size} - 0.16 * \text{Pres.} * \text{Mois. Cont.} + 0.14 \text{ Par. Size} * \text{Mois. Cont.} - 0.12 \text{ Pres.* Par. Size} * \text{Mois. Cont} \quad (5)$$

4. Conclusion

The densification parameters of sub-bituminous coal of Nigeria origin has been optimized in this study. The particle size has the largest main effect on the physicochemical properties of the coal briquette. The physicochemical properties of the coal briquettes improved with decrease in particle size. The optimum densification parameters were pressure of 56 MPa, 10% moisture content, and 0.5 mm particle size. The maximum drop to fracture, water-resistance index, and cold crushing strength of the coal briquette was 170 times/2m, 97.78%, and 7.94 MPa, respectively. Mathematical models were generated to obtain the drop to fracture, cold crushing strength, and water resistance through the particle size, moisture content,

and compacting pressure using MINITAB optimizer. The densification of the sub-bituminous coal depends mostly on the particle size of the fines. The process of optimization discussed in the study can be further applied in analyzing and optimizing other process parameters of briquettes in general.

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