Optimization of Process Variables for Briquetting of Biochar from Corn Stover

Wenqiao Jiao,[a] Lope Galindo Tabil,[b] Mingjin Xin,[a] Yuqiu Song,[a,]* Bowen Chi,[a] Liyan Wu,[a] Tianyou Chen,[c] Jun Meng,[d] and Xuewei Bai [a]

Instead of compressing biomass into briquettes, this study considers the compression of biochar. Densification is necessary for biochar to increase bulk density for convenience of handling, transportation, and storage. Response surface methodology was employed, and briquetting of biochar from corn stover was carried out in this study to investigate the effects of moisture content (at levels of 16, 17.6, 20, 22.4, and 24%), pressure (at levels of 21.5, 25, 30, 35, and 38.5 MPa), and residence time (at levels of 4, 6.4, 10, 13.6, and 16 s), on crushing resistance, dimensional stability of briquettes, and specific energy consumption of briquetting. The results showed that the effects of the variables on each evaluation index were significant ($P < 0.01$), the influence order was obtained, and the regression models are set up. The optimum condition for the briquetting process was moisture content of 18.5%, pressure of 38.5 MPa, and residence time of 4 s, giving mean values of the briquette crushing resistance of 49.9 N, dimensional stability of 93.8%, and specific energy consumption of briquetting of 4.41 MJ/t, respectively. The errors between the predicted values and the experimental values are all less than 5%.

Keywords: Biochar from corn stover; Densification; Dimensional stability; Specific energy consumption; Crushing resistance

Contact information: a: College of Engineering, Shenyang Agricultural University, Shenyang 110866, China; b: Department of Chemical and Biological Engineering, University of Saskatchewan, 57 Campus Drive, Saskatoon, SK S7N 5A9, Canada; c: College of Biological and Agricultural Engineering, Jilin University, Changchun 130025, China; d: Liaoning Biochar Engineering & Technology Research Center, Shenyang Agricultural University, Shenyang 110866, China;
** Corresponding author: songyuqiu@syau.edu.cn*

INTRODUCTION

According to the data of the National Bureau of Statistics, the total production of corn stover in China in 2018 was 3.09×10^8 t. Straw carbonized into biochar was considered as one of the attractive options to utilize the large amounts of agricultural waste (Chen *et al*. 2011) and avoid environmental pollution caused by straw open burning. Straw biochar can be extensively utilized, such as, returned to the field to improve soil physical and chemical properties and increase crop yield (Spokas *et al*. 2009; Laird *et al*. 2010); used as adsorbent for water pollution treatment (Sizmur *et al*. 2017); and employed as a new renewable clean energy to replace fossil fuels, since the emission of PM2.5 (particulates < 2.5 µm in diameter) in residential burning of biochar briquettes was much lower than that of raw fuels (Demirbas 2009; Sotannde *et al*. 2010; Sun *et al*. 2019).

However, biochar is dusty and of low bulk density (Li *et al*. 2017; Yan *et al*. 2018). Hence, densification is necessary for convenience of subsequent handling, transportation, and storage. Bazargan *et al*. (2014) used biochar (C content of 81.4% and ash of 3%) from palm kernel shell as the feedstock to explore the effects of compaction pressure, moisture content, particle size, and residence time on the tensile crushing strength, impact resistance, and water resistance of the briquettes; it was found that the tensile crushing strength of the briquettes increased from less than 40 kPa to more than 800 kPa in the weakest (longitudinal) orientation after adding starch as binder. Hu *et al*. (2016) reported that the optimum pelletizing conditions of compressing of bio-char from woody shavings at 550 to 650 °C were 35% of moisture content and 128 MPa of pressure, with the use of lignin as binder. The compressive strength of the pellet ranged from 0.85 to 16 MPa under different processing conditions. Chen *et al*. (2016) conducted single-factor experiments of densification to biochar from corn stover, and the results showed that the optimal condition for briquetting was a moisture content of 18% to 22%, pressure of 60 to 80 MPa, and residence time range of 5 to 11 s. The crushing resistance under optimal parameters were 24 to 30 N. It can be concluded that the compaction characteristics of biochar from various materials differ significantly, and that few studies have reported on the interaction of variables for the briquetting process of biochar from corn stover (BFCS).

Studies have been carried out on densification of biomass, and the quality of briquette has been found to be affected by feedstock properties and densification processes, pretreatment, and the use of additives (Tooyserkani *et al*. 2013; Tumuluru *et al*. 2015; Gong *et al*. 2015; Wu *et al*. 2015; Tilay *et al*. 2015; Kirsten *et al*. 2016). The principal factors affecting the quality of biomass pellets/briquettes include, among others, pressure; particle size; die temperature; moisture content; and residence time (Mani *et al*. 2006a; Biswas *et al*. 2014; Kazuei and Toru 2014; Garcia-Maraver *et al*. 2015; Whittaker and Shield 2017; Xin *et al*. 2017).

Since heating may result in chemical property variation of biochar and increase of energy consumption of the process, in this study, moisture content, pressure, and residence time were taken as the experimental factors, response surface methodology was employed and briquetting of BFCS was carried out to investigate the effect of the factors on crushing resistance, dimensional stability of biochar briquettes and specific energy consumption, which is important for massive production from the standpoint of economy, to determine the optimum conditions of the briquetting process.

EXPERIMENTAL

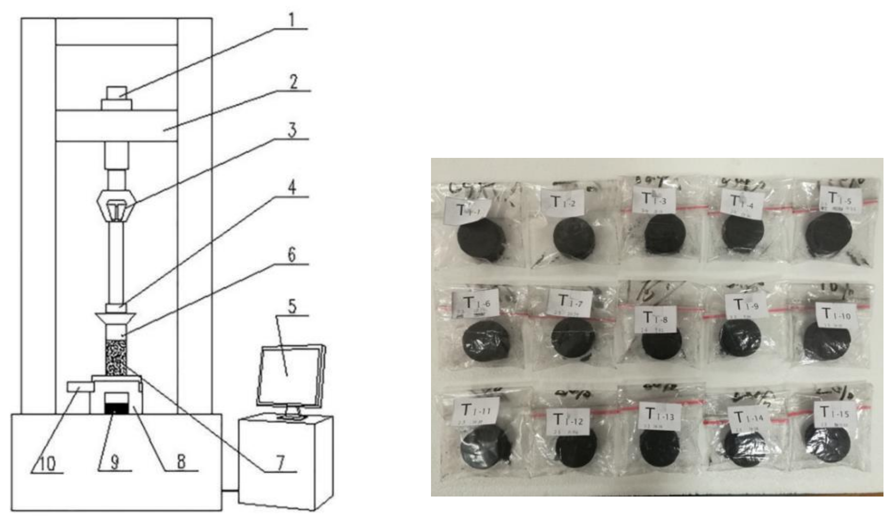
This experiment was conducted in the Laboratory of Materials of Shenyang Agricultural University (41°49′N, 123°33′E) in December 2017. The room temperature was 8 °C, and the relative humidity was 42%.

Materials

The biochar samples were obtained by pyrolysis of corn stover at about 350 °C under closed and low oxygen conditions. It had a carbon content of 45.5% and ash content of 31.4%. Its bulk density was 360 kg/m^3. The multipoint BET specific surface area was 25.8 m^2/g, and pH value was 8.4. It was dried in a far-infrared dryer (HY-1B, TonliXinda Instrument Factory, China), and then the sample was adjusted into five moisture contents (16, 17.6, 20, 22.4, and 24%) and stored in sealed barrels for 48 h. All moisture contents are expressed in % wet basis.

Densification Process

A briquetting die assembly consisting mainly of a cylindrical cartridge and a ram was designed and mounted on a computer controlled electronic universal testing machine (WDW-200, Jinan Shijin Group Co., Ltd., China) for the compression study of biochar (Fig. 1a). Before compression, 50 g BFCS was added into the cylindrical cartridge with an inner diameter of 50 mm and height of 200 mm. The pressure head compressed the BFCS to a preset pressure at the speed of 60 mm/min (Peleg and Moreyra 1979) and the specimen was kept under the specific pressure for a set residence time. The sliding gate was then pulled out, and the formed biochar briquette (Fig. 1b) was pushed out of the cartridge. The force-displacement data with densification were recorded into the computer, and five briquettes were made under each condition.



(a) **(b)**

Fig. 1. Experimental system for biochar densification; (a) the experimental system: (1) sensor, (2) beam, (3) fixture, (4) compression head (5) computer, (6) cartridge, (7) biochar, (8) support, (9) biochar briquette, (10) sliding gate; (b) the formed biochar briquettes.

Measurement of Evaluation Indices

Crushing resistance

The crushing resistance of the briquette was determined using a universal testing machine (3344R4161, Instron Corp., Canton, OH, USA). The briquette was compressed in the radial direction (Fig. 2a) because it was weaker in that direction (Kaliyan and Morey 2009). The stroke of the machine was set as 0 to 40 mm, as the diameter of the briquette was 50 mm. In each trial, the loading program was started, and the briquette was pressed from 0 mm to 40 mm until it was completely destroyed. The system unloaded automatically and the ram moved back. The curve (Fig. 2b) between the deformation (displacement) of biochar briquette and the corresponding force was recorded into the computer. The peak of the curve (maximum load, N) was taken as the crushing resistance.

Dimensional Stability

The dimension of the briquette during storage may change by stress relaxation. The diameter and height of biochar briquettes were measured immediately after briquetting and after having been sealed and stored for 72 h at room temperature. Its volume was calculated separately. The dimension stability of biochar briquette was calculated using Eq. 1,

$$DS = \left(1 - \frac{V_t - V_0}{V_0}\right) \times 100\% \quad (1)$$

where *DS* is the dimensional stability of the briquette (%), V_t is the volume of the briquette after 72 h(m^3), and V_0 is the volume of the briquette immediately after densification (m^3).

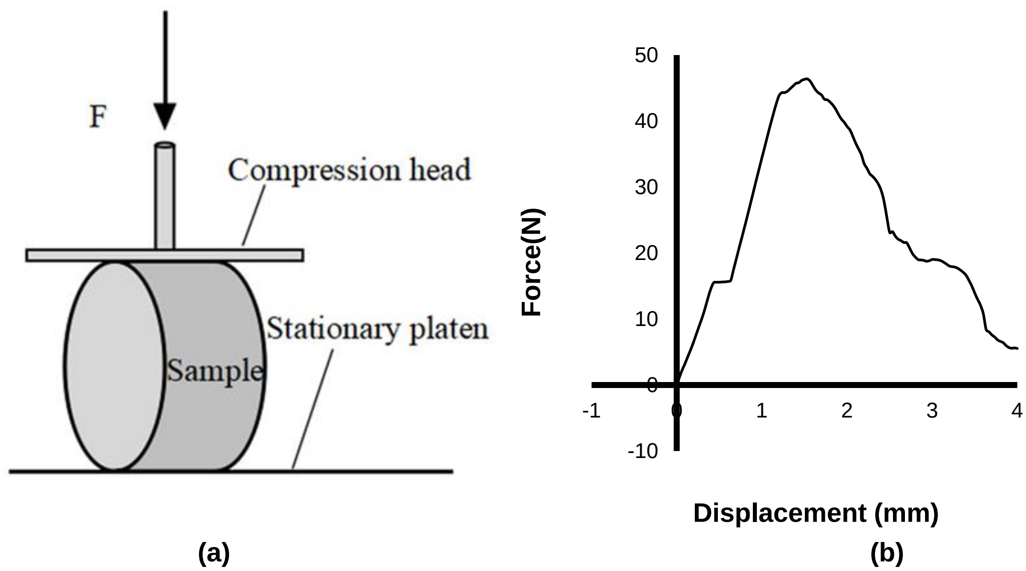


Fig. 2. Crushing resistance testing. (a) Loading direction; (b) curve of force with displacement

Specific Energy Consumption

The specific energy consumption (E_m) of briquetting was calculated with Eq. (2) based on force-displacement data recorded by the computer controlled electronic universal testing machine during densification.

$$E_m = \frac{W}{m} = \frac{\int_0^S F \times S d_S}{m} \quad (2)$$

where *W* is the energy consumption (kJ), *m* is the mass of densified biochar briquette (kg), *F* is the force (N), and *S* is the displacement (m).

Experimental Design

A five-level-three-factor central composite rotatable design (CCRD) was adopted. The variables for densification characteristics of BFCS were moisture content (16, 17.6, 20, 22.4, and 24%), pressure (21.5, 25, 30, 35, and 38.5 MPa), and residence time (4, 6.4, 10, 13.6, and 16 s); the ranges were determined by trial experiments. The evaluation indexes were the crushing resistance (Y_1) of biochar briquette, dimensional stability (Y_2), and the specific energy consumption (Y_3). Table 1 shows the five coded levels (−1.682, −1, 0, +1, +1.682) of independent factors (X_i) and the experimental design.

Statistical Analysis and Optimization

Since each of the three indicators had its own optimal range of variables, comprehensive optimization was conducted, and weight coefficients were given to crushing resistance, dimensional stability of biochar briquette, and specific energy consumption of briquetting as 0.5, 0.3, and 0.2, respectively, based on comprehensive analysis of the importance degree of densification index for transportation, subsequent utilization, and production cost. Design Expert software (V. 8.0.6, STAS-EASE Inc., Minneapolis, MN, USA) was employed to generate response surfaces and contour plots, analyze experimental data, and conduct multi-objective optimization. The fitting quality of the model was evaluated by the determination coefficients and analysis of variance (ANOVA). The quadratic polynomial equation of the quality of biochar briquettes, the response of densification process (Y), as a function of the independent variables and their interaction could be established by utilizing Eq. 3,

$$Y = \beta_0 + \sum_{i=1}^{3} \beta_i X_i + \sum_{i=1}^{3} \beta_{ii} X_i^2 + \sum_{i=1}^{2} \sum_{j=i+1}^{3} \beta_{ii} X_i X_j \tag{3}$$

where Y is the response value (crushing resistance, dimensional stability and energy consumption), X_i is the independent factor, β_0 is the intercept, β_i is the first order model coefficient, β_{ii} is the quadratic coefficient for the variables i and β_{ij} is the linear model coefficient for the interaction between variable i and j.

Table 1. Coding of Factors and Levels for the Central Composite Rotatable Design

Variables	Symbols	Coded Factor Levels				
		-1.682	-1	0	1	1.682
Moisture content (%)	X_1	16.0	17.6	20.0	22.4	24.0
Pressure (MPa)	X_2	21.5	25	30	35	38.5
Residence time (s)	X_3	4.0	6.4	10	13.6	16.0

RESULTS AND DISCUSSION

Development of the Regression Model Equation

According to the results of single factor experiments, the CCRD experiment was carried out with eight factorial points, six axial points, and nine central points in this study. The experimental results are summarized in Table 2 and analyzed using response surface methodology.

Regression model equation for crushing resistance

The crushing resistance of biochar briquette (Y_1) was tested after sealed and stored for 72 h after compression. The regression model of crushing resistance with moisture content (X_1), pressure (X_2), and residence time (X_3) was developed as Eq. 4. The ANOVA significance test results are presented in Table 3.

$$Y_1 = 42.39 - 2.17X_1 + 1.04X_2 + 2.66X_3 + 1.01X_1X_2 + 0.20X_1X_3 - 6.06X_2X_3 - 0.82X_1^2 - 1.18X_2^2 - 1.11X_3^2 \tag{4}$$

Table 2. Crushing Resistance, Dimensional Stability, and Specific Energy Consumption of Biochar from Corn Stover

Run	Factor Levels			Crushing Resistance (Y_1/N)	Dimensional Stability (Y_2/%)	Specific Energy Consumption (Y_3/MJ/t)
	Moisture Content (%)	Pressure (MPa)	Residence Time (s)			
1	-1	-1	-1	33.18	96.4	6.66
2	1	-1	-1	26.54	95.8	6.82
3	-1	1	-1	45.66	94.7	5.04
4	1	1	-1	43.01	92.2	5.00
5	-1	-1	1	50.44	93.9	6.98
6	1	-1	1	44.55	94.2	7.19
7	-1	1	1	38.63	92.9	5.28
8	1	1	1	36.83	91.9	5.38
9	-1.682	0	0	43.02	95.8	5.88
10	1.682	0	0	35.53	94.7	6.13
11	0	-1.682	0	36.83	95.4	7.52
12	0	1.682	0	39.70	92.4	4.50
13	0	0	-1.682	34.18	95.8	5.88
14	0	0	1.682	42.69	92.8	6.36
15	0	0	0	42.83	95.1	6.20
16	0	0	0	43.12	94.6	6.17
17	0	0	0	41.25	94.8	6.08
18	0	0	0	41.58	94.5	6.1
19	0	0	0	43.10	95.2	6.08
20	0	0	0	42.95	95	6.20
21	0	0	0	42.26	94.7	6.18
22	0	0	0	41.95	94.8	6.14
23	0	0	0	42.78	94.9	6.10

Table 3. Analysis of Variance for Response Surface Quadratic Model of Briquette Crushing Resistance

Source	Sum of Squares	Degree of Freedom	Mean Square	F-value	p-value Prob > F	Significance
Model	529.69	9	58.85	70.20	< 0.0001	* *
X_1	64.05	1	64.05	76.40	< 0.0001	* *
X_2	14.86	1	14.86	17.73	0.0010	* *
X_3	96.87	1	96.87	115.54	< 0.0001	* *
X_1X_2	8.16	1	8.16	9.73	0.0081	* *
X_1X_3	0.32	1	0.32	0.38	0.5474	
X_2X_3	293.79	1	293.79	350.40	< 0.0001	* *
X_1^2	10.63	1	10.63	12.68	0.0035	* *
X_2^2	21.94	1	21.94	26.17	0.0002	* *
X_3^2	19.75	1	19.75	23.56	0.0003	* *
Residual	10.90	13	0.84			
Lack of Fit	7.05	5	1.41	2.93	0.0857	
Pure Error	3.85	8	0.48			
Cor. Total	540.59	22				

Note: * * represents extremely significant (P<0.01); * represents significant (P<0.05).

The crushing resistance of the BFCS briquette was affected by the selected factors according to quadratic relationships. The test results of the regression model showed that F = 70.20 > $F_{0.01}(9,13)$ = 3.45, P < 0.0001, indicating that the regression model was highly significant. The coefficient of determination was 0.97 (R^2 = 0.97), indicating that about 97% of the total variation in the results could be explained by the model. And the *Lack of Fit* = 2.93 < $F_{0.01}(5,8)$ = 3.69, P = 0.086 > 0.05, indicated that the Lack of Fit was not significant compared to pure error; therefore, the estimating model fit with the experimental data. The effects of the three process variables on the crushing resistance were significant (P < 0.01), and the results were consistent with that of most researches on densification behaviors of materials other than biochar (Al-Widyan *et al*. 2002; Kaliyan and Morey 2009; Hu *et al*. 2016), and the influencing order is: residence time > moisture content > pressure. The interaction of moisture content and pressure and the interaction of pressure and residence time have significant effects on the crushing resistance of briquettes (P < 0.01). The final regression equation is given as Eq. 5 after eliminating insignificant terms at P=0.05.

$$Y_1 = 42.39 - 2.17X_1 + 1.04X_2 + 2.66X_3 + 1.01X_1X_2 - 6.06X_2X_3 - 0.82X_1^2 - 1.18X_2^2 - 1.11X_3^2 \quad (5)$$

Regression model for briquette dimensional stability

The regression model for dimensional stability (Y_2) of the BFCS briquette was obtained from the experimental results as shown in Eq. 6.

$$Y_2 = 94.85 - 0.41X_1 - 1.00X_2 - 0.82X_3 - 0.04X_1X_2 + 0.30X_1X_3 + 0.25X_2X_3 - 0.048X_1^2 - 0.43X_2^2 - 0.29X_3^2 \quad (6)$$

Table 4. Analysis of Variance for Response Surface Quadratic Model of Briquette Dimensional Stability

Source	Sum of Squares	Degree of Freedom	Mean Square	F-value	P-value Prob > F	Significance
Model	32.00	9	3.56	33.58	< 0.0001	* *
X_1	2.34	1	2.34	22.07	0.0004	* *
X_2	13.63	1	13.63	128.75	< 0.0001	* *
X_3	9.26	1	9.26	87.45	< 0.0001	* *
X_1X_2	1.28	1	1.28	12.09	0.0041	* *
X_1X_3	0.72	1	0.72	6.80	0.0217	* *
X_2X_3	0.50	1	0.50	4.72	0.0489	* *
X_1^2	0.036	1	0.04	0.34	0.5690	
X_2^2	2.93	1	2.93	27.69	0.0002	* *
X_3^2	1.32	1	1.32	12.46	0.0037	* *
Residual	1.38	13	0.11			
Lack of Fit	0.95	5	0.19	3.62	0.0524	
Pure Error	0.42	8	0.05			
Cor. Total	33.38	22				

At a significance level of P = 0.05, significance analysis and ANOVA were carried out for the regression equation, and the analysis of variance is shown in Table 4. The regression equation model's F value equaled 33.58 > $F_{0.01}(9,13)$ = 3.45, P < 0.0001. These findings indicated that the regression model was extremely significant. The coefficient of determination was 0.93 (R^2 = 0.93), and the *Lack of Fit* = 3.62 < $F_{0.01}(5,8)$ = 3.69, P = 0.0524 >0.05; therefore, the estimating model fit the experimental data adequately and can be used to predict the dimensional stability of the biochar briquette. The influence of the independent variables on dimensional stability was highly significant (P < 0.01), and the influence order of the independent variables was: pressure > residence time > moisture content. Besides, the interaction of moisture content and pressure, the interaction of moisture content and residence time and the interaction of pressure and residence time have significant effect on dimensional stability (P < 0.05). Kaliyan *et al*. (2009) also reported that dimensional stability was mainly dependent on feedstock moisture content and compression pressure. The final regression model is obtained as Eq. 7 after ignoring insignificant terms at P=0.05:

$$Y_2 = 94.85 - 0.41X_1 - 1.00X_2 - 0.82X_3 - 0.04X_1X_2 + 0.30X_1X_3 + 0.25X_2X_3 - 0.43X_2^2 - 0.29X_3^2 \quad (7)$$

Regression model for specific energy consumption

The regression model for specific energy consumption during briquetting of BFCS was obtained as Eq. 8 according to analysis of the experimental data, and the analysis of variance is shown in Table 5.

$$Y_3 = 6.14 + 0.062X_1 - 0.88X_2 + 0.16X_3 - 0.039X_1X_2 + 0.024X_1X_3 - 8.75\times10^{-3}X_2X_3 - 0.046X_1^2 - 0.045X_2^2 - 5.756\times10^{-4}X_3^2 \quad (8)$$

Table 5. Analysis of Variance for Response Surface Quadratic Model of Specific Energy Consumption during Briquetting

Source	Sum of Squares	Degree of Freedom	Mean Square	F-value	P-value Prob > F	Significance
Model	11.06	9	1.23	595.15	< 0.0001	* *
X_1	0.053	1	0.053	25.65	0.0002	* *
X_2	10.60	1	10.60	5131.54	< 0.0001	* *
X_3	0.33	1	0.33	158.98	< 0.0001	* *
X_1X_2	0.012	1	0.012	5.82	0.0314	* *
X_1X_3	0.0045	1	0.0045	2.19	0.1631	
X_2X_3	0.0006	1	0.0006	0.30	0.5952	
X_1^2	0.034	1	0.034	16.58	0.0013	* *
X_2^2	0.032	1	0.032	15.34	0.0018	* *
X_3^2	0.0005	1	0.0005	0.26	0.6220	
Residual	0.027	13	0.0021			
Lack of Fit	0.0068	5	0.00135	0.54	0.7437	
Pure Error	0.020	8	0.0025			
Cor. Total	11.09	22				

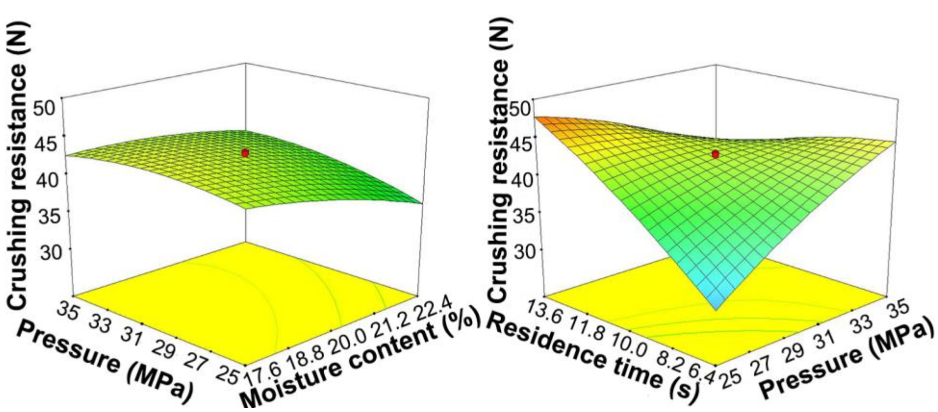
At a significance level of P = 0.05, significance analysis and ANOVA were carried out for the regression equation, and the analysis results are shown in Table 5. The regression equation gave F = 595.15 > $F_{0.01}(9,13)$ = 3.45, P < 0.01, indicating that the regression model was highly significant. The complex correlation index R^2=0.99, and the *Lack of Fit* = 0.54 < $F_{0.01}(5,8)$ = 3.69, P = 0.07 > 0.05; therefore the estimating model fit the experimental data adequately and could be used to predict the specific energy consumption of the compaction. The influence of the independent variables on specific energy consumption was highly significant (P < 0.01), and the influence order of factors is: pressure > moisture content > residence time. In addition, the interaction of moisture content and pressure had significant effect on specific energy consumption (P < 0.05). The investigation of Pampuro *et al*. (2013) showed that pressure had more significant effect on energy consumption of compost densification than residence time as well. The final regression model is obtained by Eq. 9 after eliminating the non-significant term at P=0.05.

$$Y_3 = 6.14 + 0.062X_1 - 0.88X_2 + 0.16X_3 - 0.039X_1X_2 - 0.046X_1^2 - 0.045X_2^2 \quad (9)$$

Response Surface Analysis of Interaction of Process Variables on Index

Effect of interaction on crushing resistance

The results in Table 3 show that interactions between the variables had a highly significant effect on crushing resistance of the briquette. It can be concluded from Fig. 3a that the crushing resistance of briquette slightly increased with the increase of pressure at any designed level of moisture content when the residence time was 10 s, and it decreased with the increase of moisture content when the pressure was constant. It can be observed from Fig. 3b that, when the moisture content was 20%, the crushing resistance increased with the increase of residence time at the lowest pressure; and it decreased with the increase of residence time while the pressure was at a relatively higher level. Within the studied range of residence time, the crushing resistance increased linearly with pressure at lower level of residence time, and it decreased with increase of pressure under a longer residence time. According to the extreme value theory of multivariate function, the partial derivative of the regression equation of the crushing resistance of biochar briquette was obtained. The optimal technological conditions were as follows: water content of 16%, pressure of 21.5 MPa, and residence time of 16 s, and then the crushing resistance of biochar briquette was predicted to be 59.38 N.



a) Moisture content and Pressure **b)** Pressure and Residence time

Fig. 3. Response surface plot representing effect of pressure and residence time on crushing resistance of biochar briquette (other factor is constant at zero levels). The interactions between (a) moisture and pressure; (b) pressure and residence time

Effect of interaction on briquette dimensional stability

The effects of interaction of factors on dimensional stability of biochar briquette are shown in Fig. 4. When the residence time was 10 s, the dimensional stability of the biochar briquette decreased with an increase of pressure at any designed level of moisture content; within the studied range of pressure, it was not highly relevant to moisture content at a lower level of pressure, and it decreased with an increase of moisture content under higher pressure. As shown in Fig. 4b, when the pressure was 30 MPa, the dimensional stability decreased with the increase of residence time as the moisture content was kept constant; and it was not highly relevant to variation of moisture content under longer residence time. Figure 4c presents the effects of pressure and residence time on dimensional stability of biochar briquette at a moisture content of 20%. It can be concluded that the dimensional stability decreased with the increase of residence time at a constant pressure; and it also decreased with the increase of the pressure at a constant residence time. Al-Widyan *et al.* (2002), who investigated the stability (relaxed density) of olive cake briquettes under varying pressure, moisture content, and residence time, reported a similar result that the maximum residence time for olive cake densification should not exceed 5 s.

The partial derivative of the regression model of the dimensional stability of biochar briquette was obtained based on the extreme value theory of multivariate function, and the optimal technological conditions for biochar briquetting were found to be a water content of 16%, pressure of 25.6 MPa, and residence time of 4 s, when the predicted dimensional stability of biochar briquette was 97.4%.

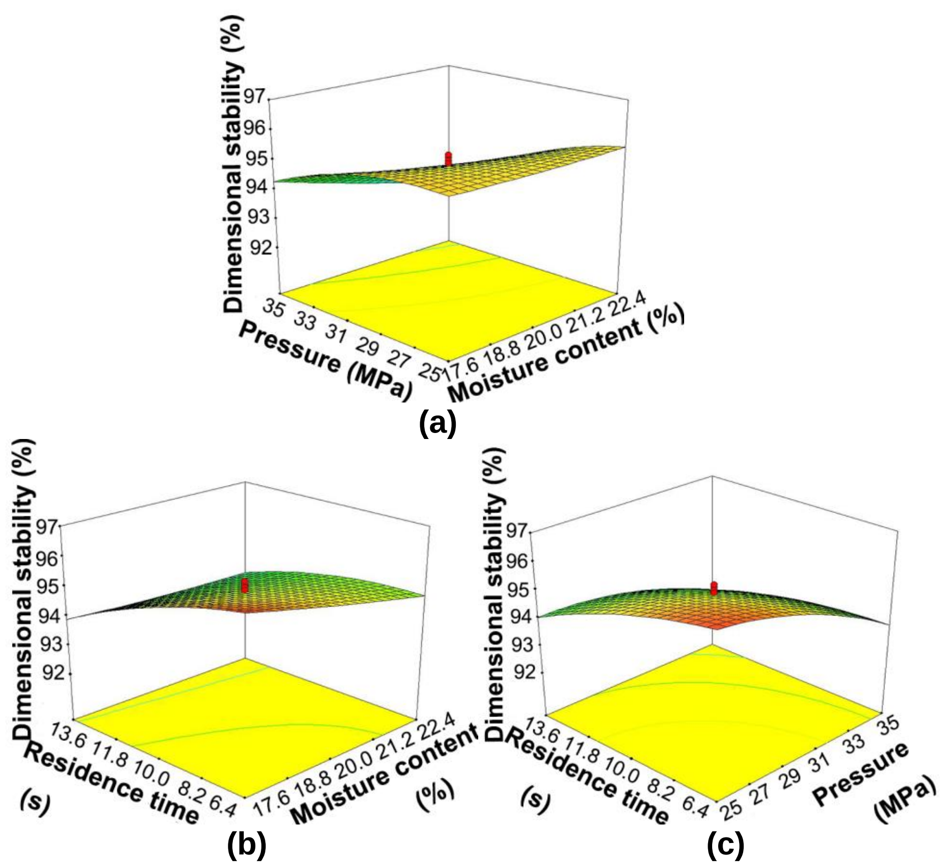


Fig. 4. Response surface plot for dimensional stability; other factors are constant at zero levels. The interactions between (a) moisture and pressure, (b) moisture and residence time and (c) pressure and residence time

Effect of interaction on specific energy consumption

The interaction of pressure and residence time significantly impacted specific energy consumption (P = 0.03< 0.05) (Table 4). Figure 5 shows that when the residence time was 10 s, the specific energy consumption of the briquetting decreased with the increase of pressure at any level of introduced moisture content; and within the studied range of pressure, it was not highly relevant to the increment in moisture content. The partial derivative of the regression model of specific energy consumption was obtained based on the extreme value theory of multivariate function. The optimal technological conditions for briquetting on the point of specific energy consumption within the range of tested levels of factors were as follows: moisture content of 24%, pressure of 38.5 MPa, residence time of 4 s, and then the specific energy consumption for biochar briquetting was predicted to be 4.06 MJ/t. Mani *et al*. (2006b) reported a high specific energy consumption for briquetting of corn stover as 6 to 15MJ/t at conditions of moisture content of 5 to 15%, and pressure of 5 to 15 MPa. The reason might be the properties of the material and the conditions for the briquetting, and the mechanism needs to be further studied.

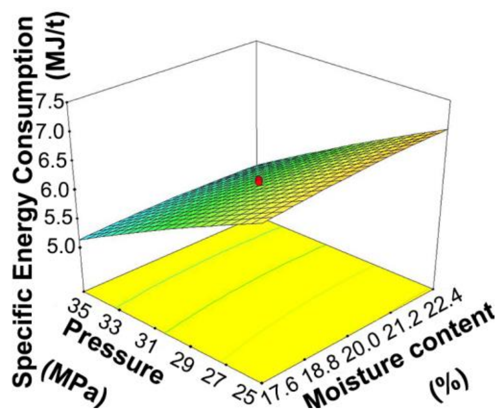


Fig. 5. Response surface plot representing effect of pressure and moisture content on specific energy consumption of briquetting (other factor is constant at zero levels)

Process Optimization and Verification Experiment

The goal parameters of crushing resistance and dimensional stability were set to be maximized; the energy consumption during briquetting was minimized; and the weight coefficients were 0.5, 0.3, and 0.2, respectively (as discussed in the previous section). The optimum conditions are obtained, from calculation with Design–Expert 8.0.6 software, to be 18.6% of moisture content, 38.5 MPa of pressure, and 4 s of residence time, which gives the briquette crushing resistance of 50.6 N, dimensional stability of 92.8%, and specific energy consumption of 4.27 MJ/t.

A verification experiment was carried out, and considering the operability of the experiment, the optimum conditions were adjusted to be 18.5% of moisture content, 38.5 MPa of pressure, and 4 s of residence time. The experiment was replicated three times. The experimental results and predicted values are presented in Table 6. It can be concluded that under optimized conditions, the average values of index were crushing resistance of 49.9 N, the dimensional stability of 93.8%, and the specific energy consumption of 4.41 MJ/t; the errors were 2.1%, 1.4%, and 3.1%, respectively. The response surface model and the experimental design were shown to be reliable and repeatable.

The above research was based on the biochar samples of 45.5% carbon and 31.4% ash. Since the principal content of the biochar from slow pyrolysis differ considerably

(Wang *et al*. 2020) and the temperature had a significant effect on yield, constituents content of bio-char, and the densification process (Hu *et al*. 2016), further study on densification of BFCS at various pyrolysis temperatures should be carried out in order to achieve better understanding of the briquetting characteristics of the BFCS.

Table 6. Validation of Model Adequacy

Run	Variables			Y_1 (N)			Y_2 (%)			Y_3 (MJ/t)		
	X_1 (%)	X_2 (MPa)	X_3 (s)	P	E	Error (%)	P	E	Error (%)	P	E	Error (%)
1	18.5	38.5	4.0	50.57	48.6	3.9	92.79	92.31	0.5	4.27	4.36	2.1
2					49.98	1.2		93.87	1.2		4.52	5.5
3					51.24	1.3		95.12	2.4		4.34	1.6
Average value				50.57	49.94	2.1	92.79	93.77	1.4	4.27	4.41	3.1

Note: P is predicted value; E is experimental value

CONCLUSIONS

1. The effects of the three process variables, moisture content, pressure, and residence time, were found to be extremely significant ($P < 0.01$) relative to crushing resistance, dimensional stability, and specific energy consumption for briquetting of biochar from corn stover. The regression models of crushing resistance, dimensional stability, and specific energy consumption were obtained, and the coefficient of determination were 0.97, 0.93, and 0.99 ($R^2 > 0.9$), respectively.
2. The interactions of moisture content and pressure, and of residence time and pressure, were extremely significant on crushing resistance of biochar briquette ($P < 0.01$); the interactions of moisture content and pressure, of residence time and moisture content, and of residence time and pressure, were extremely significant on dimensional stability of the compressed briquette ($P < 0.01$); the interaction of moisture content and pressure was extremely significant on specific energy consumption of briquetting ($P < 0.01$).
3. The optimum conditions for briquetting, based on comprehensive optimization and setting weight coefficients, 0.5, 0.3, and 0.2, to indicators of crushing resistance, dimensional stability, and specific energy consumption, respectively, were 18.5% of moisture content, 38.5 MPa of pressure, and 4 s of residence time, resulting in an average briquette crushing resistance of 49.9 N, dimension stability of 93.8%, and specific energy consumption of 4.41 MJ/t.

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