Optimization of binder addition and compression load for pelletization of wheat straw using response surface methodology

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Abstract: Densification is required for efficiently handling and transporting biomass as feedstock for biofuel production. Binders can enhance straw pellet strength and improve the pellet performance. The present investigation aimed to optimize binders and compression load for wheat straw pelletization using a single pelleting unit. Response surface methodology was employed by using a four-factor, five-level central composite design with wood residue (%, w/w), bentonite (%, w/w), crude glycerol (%, w/w), and compression load (N) as process parameters. The pellet tensile strength, specific energy consumption of pelleting, and pellet density were the response variables. The higher heating value, ash content of the pellet product and the cost of the feedstock were also considered in optimizing binder addition. The developed model fitted the data and was adequate for binder analysis and optimization. Wheat straw pellet, with the addition of 30% wood residue, 0.80% bentonite, and 3.42% crude glycerol, in addition to 4 000 N of compressive load, was identified as optimal with good performance of pellet tensile strength (1.14 MPa), specific energy consumption (32.6 kJ/kg), and pellet density (1 094 kg/m^3) as well as low ash content (6.13%) and high heating value (18.64 MJ/kg). Confirmation tests indicated high accuracy of the model.

Keywords: biomass, wheat straw pellet, binder, wood residue, bentonite, crude glycerol, RSM, compression load
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1 Introduction

Agricultural residues, the largest available biomass resource in the world for bioenergy production[1], are environmental-friendly feedstock with huge potential for sustainable energy production. However, agricultural straw has low bulk density which does not make it easy to handle or transport. Logistics cost is often high due to the great distance between the origin of biomass and the location for energy production[2]. Densification of biomass into pellets can significantly reduce the cost of handling, transportation, and storage[3]. The volumetric calorific value and physical properties can be improved, and the size and shape of biomass can be made uniform

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through densification[4]. The lack of natural binder in the agricultural straw is one of the important reasons for poorly formed pellets which are often dusty, difficult to handle, and costly to manufacture[4]. Some pretreatment methods have been explored to improve the durability and strength of biomass pellets[5-8]. By controlling the processing method and related parameters, high quality pellets could be produced. Binder addition is a method employed in this study. Optimization of binder addition for the pelletization process is one of the most important steps in the development of an efficient and cost-effective energy conservation strategy using biomass.

Lignin is one kind of natural binder for biomass densification[9]. Wood residue with high lignin content can act as a potential binder for wheat straw densification. The heating value of wheat straw pellets can be potentially improved with the addition of wood residue because of its higher heating value. It is environmentally friendly to recycle wood residue, which also can solve a disposal problem by using them to make biofuel[10]. Ståhl and Berghel[10] investigated the effect of mixing sawdust and rapeseed cake to make pellets. They reported that the durability of the pellets increased proportionally with an increase in the amount of sawdust. Kaliyan and Morey[9] suggested that biological binders such as sawdust (more than 20% by weight) can help improve the quality of the densified product.

Colloidal hydrated magnesium aluminum silicate clay or bentonite had been used for making durable feed pellets[11]. Bentonite added at a rate of 2.6% can reduce dust generation of feed pellets[12]. Bentonite has been reportedly used at an inclusion rate of 5.0% to produce alfalfa pellets which can improve pellet durability[11].

Crude glycerol, a byproduct of the transesterification process in the biodiesel industry, is an additive considered in this study. One kilogram of crude glycerol is co-produced for every 10 kg biodiesel produced[13]. There is a positive expectation for glycerol's production with the spread of biodiesel production plants[14] so that crude glycerol supply may exceed its demand. Crude glycerol is an energy source[15] with a higher heating value compared to agricultural biomass or wood residue. Glycerol could act as a lubricant during pelleting and its addition could potentially enhance the heating value of biomass pellets.

Response surface methodology (RSM) is a statistical tool based on the multivariate non-linear model, which is useful in analyzing various parameters affecting the process[16]. Process optimization is based on the simultaneous combination of all goals for the responses and factors. RSM has been widely used in development of new processes and the optimization of their performance[17].

In the present study, the optimization of the addition of binders and applied compression load during wheat straw pellet production was conducted by using RSM. The objective of this study is to determine the most promising binder combination and compression load for wheat straw pelletization by maximizing pellet strength, minimizing energy consumption of pelleting, and maximizing pellet density. Heating value, ash content and binder cost is also considered.

2 Materials and methods

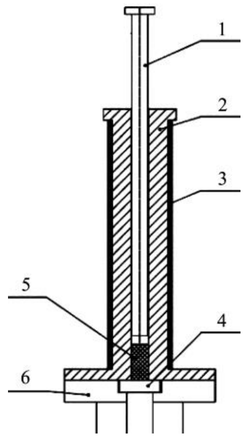
2.1 Materials and pelletization experimental setup

The details of the processes and experimental single pellet unit in these experiments are presented in an earlier paper[18]. A brief description of the process and equipment is as below. Wheat straw (*Triticum aestivum* L.) was acquired in the form of small square bales with dimensions of 0.45 m×0.35 m×1.00 m from the Central Butte area (50.83°N, 106.51°W) in Saskatchewan, Canada, which was harvested in the fall of 2011. Bentonite was obtained from Canadian Clay Products, Inc. (Wilcox, SK, Canada). Crude glycerol was acquired from Milligan Bio-Tech, Inc. (Foam Lake, SK, Canada). Wood residue was purchased in the form of soft wood shavings with 60% spruce and 40% pine from Western Wood Shavings, Inc. (Saskatoon, SK, Canada).

Wheat straw and wood residue samples were first ground using a hammer mill (Serial no. 6M13688; Glen Mills Inc., Maywood, NJ, USA) powered by a 1.5 kW electric motor which was connected to a dust collector (Model No. DC-202B, House of Tools, Saskatoon, SK, Canada) to control dust during operation, provide flowability of the ground biomass through the hammer mill, and collect the ground biomass. A screen size of 3.2 mm was used in the hammer mill. The particle size analysis of wheat straw grinds and wood residue grinds was conducted using ASABE Standard S319.3[19]. A Ro-Tap sieve shaker (W. S. Tyler Inc., Mentor, OH, USA) and U.S. sieve numbers 16, 20, 30, 50, 70, and 100 (sieve opening sizes: 1.190, 0.841, 0.595, 0.297, 0.210, and 0.149 mm, respectively) were used for the particle size analysis in 3 replicates. The mean geometric particle size of wood residue grinds and wheat straw grinds was (1.022±0.017) mm and (0.858±0.001) mm, respectively. As the geometric particle size of wood waste grinds was larger than wheat straw grinds, the geometric particle size of wood residue grinds was adjusted by changing the mass percentage on sieve no.16 by using the formula suggested in the ASABE Standard S319.3[19] to obtain the same geometric mean diameter with wheat straw grinds. The adjusted mass percentage of particles retained on each sieve size was presented in an earlier paper[18]. In brief, the average percentage of wood particles on sieve number 16 was changed from 61.77% to 41.27%, and the average percentages of wood particles on other sieves were increased slightly and accordingly. Wood residue samples were recreated by mixing grinds from each sieve with the adjusted proportion.

The moisture content of the ground material and the pretreated wood residue samples were measured using oven drying at 103±2℃ for 24 h[20] in three replicates. The average initial moisture contents of the wheat straw grinds and the softwood grinds were 7.30% (w.b.) and 6.66% (w.b.), respectively. The moisture content of wheat straw grinds and wood residue grinds were both adjusted to a range from 9% to10% by spraying water. Then the samples were kept in air-tight plastic bags for one week to allow for moisture equilibration.

Wheat straw samples were pelleted in a single pelleting unit (Figure 1) used in previous research[21-26]. In brief, the device is composed of a steel cylindrical die with an internal diameter of 6.35 mm and a length of 125 mm. A heating element maintaining the die wall temperature at 95±1℃ was wrapped on the outside of the die. A plunger connected to the upper moving crosshead of an Instron Model 3366 (Instron Corp., Norwood, MA, USA) universal testing machine compressed the sample at a speed of 50 mm/min. The cylindrical die was slip fitted into a stainless steel base with a hole matching the outer diameter of die. This steel base allowed the plunger to move straight down with no lateral movement during pelletization. Approximately 0.5 g sample was loaded into the cylindrical die for each run. Compressive force applied can be adjusted by changing the program. After compression and achieving the pre-set load, the plunger was stopped and held in position for 60 s. Then the pellet was ejected from the die at a speed of 50 mm/min using the plunger after the base plate was removed. The force-displacement curve and data was recorded using the Bluehill software (Version 2.12, Illinois Tool Works, Inc., 2010). The single pelletization process was replicated eight times for each sample.



1. Plunger 2. Cylindrical die 3. Heating element 4. Base plate
5. Pellet sample 6. Base

Figure 1 Schematic diagram of the single pellet unit

2.2 Experimental design

Based on the results of an earlier study[18], bentonite, wood residue, and crude glycerol performed well as binders or additives. Bentonite is good at improving pellet strength and reducing energy consumption. Wood residue can increase the pellet strength, density, and heating value of wheat straw pellets. Crude glycerol can significantly increase the heating value of the straw pellet. So, in this work these three binders were combined as a mixed binder to improve pellet quality. In addition, the compression load for pelleting was also considered to be a factor and then the interaction between adding binder and energy consumption would be observed more clearly. Pellet tensile strength, specific energy consumption of pelleting, and pellet density were chosen to be responses because strength, energy, and density are three of the most important characteristics for evaluating pellet quality. Response surface methodology (RSM), using a central composite factorial design (CCD) with four factors, six central points, and eight axial points, was used to design the experimental trials. Design-Expert software, version 7.0 (Stat-Ease Inc., Minneapolis, MN, USA) was used to conduct the experimental design and the optimization. A total of 30 experiments were performed. The order of the experiments was randomized. Factors and their respective levels are summarized in Table 1.

Table 1 Experimental codes and actual levels of the independent variables

Independent variable	Symbol	Code and level		
		-1	0	1
Wood residue/%	A	10	20	30
Bentonite/%	B	1	2	3
Glycerol%	C	2	4	6
Compression load/N	D	2 000	3 000	4 000

2.3 Mixing binders with wheat straw

Binders were directly mixed into wheat straw grinds according to the experimental design except crude glycerol. Crude glycerol was heated to above 160℃ using a hot plate and stirred with a mini size magnetic stirrer at 220 r/min to reduce its viscosity before spraying it into wheat straw grinds. In order to achieve uniform distribution of each material, the mixing was carried out for 15 min; after mixing, the samples were stored in air-tight bags. Subsequent mixing was carried out every 12 h for at least 72 h.

2.4 Tensile strength of pellets

The strength and durability of the densified products would help optimize the material, pretreatment, and equipment to produce high quality densified products[5]. Pellet tensile strength was determined in lieu of pellet durability because of the limited number of pellets produced in single pelleting experiments. The tensile strength of the single pellet was evaluated using the diametral compression test[21, 25]. A Dremel rotary tool (Robert Bosch Tool Corp., Racine, WI, USA) with a diamond cutting wheel bit[25] was used to cut the pellet due to the high strength of the binder-straw pellet. Pellets were firstly cut diametrically into approximately 2 mm thick specimens and placed longitudinally at the centre of the base plate for testing. The base plate and plunger of the Instron were padded with blotting paper. Each of the specimens was pressed by the plunger of the Instron at a speed of 1 mm/min. The force and displacement data were recorded and the fracture force was determined by choosing the point that the specimen was split apart into two semi-circular segments. Tensile strength of the pellet was determined and calculated using Equation (1)[21, 25]. This test was replicated eight times for each sample.

$$\sigma_x = \frac{2F}{\pi dl} \tag{1}$$

where: σ_x is tensile (horizontal) stress, Pa; F is load at fracture, N; d is specimen diameter, m; l is specimen thickness, m.

2.5 Specific energy consumption

Energy consumption was calculated by integrating the area under the force-displacement curve recorded by the computer. Then, it was converted to specific energy values in MJ/t by dividing the pellet mass.

2.6 Pellet density

Immediately after the ejection of the pellet, its dimensions were measured using a digital caliper. The pellet mass was also measured. Pellet density was calculated using mass divided by pellet volume.

2.7 Model development and diagnostics

The Design-Expert software, version 7.0 (Stat-Ease Inc., Minneapolis, MN, USA) was used to create the model and analyze data. The relationship between factors (wood residue addition, bentonite addition, crude glycerol addition, and compression load) and the responses (pellet tensile strength, specific energy consumption, and pellet density) was evaluated by the best fit model suggested by the Design Expert software. The modified models were derived by excluding insignificant terms. The Model Diagnostic Plots were conducted by the Design Expert software as well.

The main indicators demonstrating the significance and adequacy of the model are: the modified models were significant; the lack of fit was insignificant; the adjusted and predicted R^2 values were within 0.2 of each other; the adequate precision was over 4[27]; and all the residuals were random. Then the model is considered to give good predictions for average outcomes. The significance of model terms was estimated based on P-values at 95% confidence level.

2.8 Optimization method

The goals for optimization were set for both responses and variables. Other factors were also taken into consideration during optimization. These objectives were input in the Design-Expert software, version 7.0 (Stat-Ease Inc., Minneapolis, MN, USA). The Design-Expert software was also used to draw the three-dimensional plot of response surfaces.

Other factors (heating value, ash content, and cost) were also considered to be minor factors that affect the final optimization. The heating value of the samples was measured in three replicates using a Parr 1281 automatic isoperibol oxygen bomb calorimeter (Parr Instrument Co., Moline, IL, USA) based upon ASTM D5865-03[28]. The total ash content was determined using AOAC standard method 942.05[29]. Information on binder costs was gathered and also considered to be a factor in optimization. The cost data of the wheat straw and binders were acquired by consulting companies selling these products.

3 Results and discussion

The results of the experimental runs are shown in Table 2. The mean pellet tensile strength varied from 0.69 to 1.24 MPa. The specific energy consumption for pelleting ranged from 18.6 to 36.6 MJ/t. Pellet density varied from 883.0 to 1 105.6 kg/m^3.

Table 2 Mean values of pellet tensile strength, specific energy consumption and pellet density during the experimental runs

Run	Wood residue /%	Bentonite /%	Crude glycerol /%	Compression load /N	Tensile strength /MPa	Specific energy consumption /MJ t^{-1}	Pellet density /kg m^{-3}
1	30	1	2	2 000	0.89	26.0	1 010
2	20	2	4	3 000	0.97	31.0	1 052
3	30	1	6	4 000	0.88	31.8	1 071
4	20	0	4	3 000	0.91	33.8	1 041
5	30	1	2	4 000	1.24	33.5	1 071
6	20	2	4	3 000	0.92	30.1	1 050
7	10	1	6	4 000	0.80	33.5	1 055
8	20	2	4	3 000	0.97	29.5	1 062
9	20	2	8	3 000	0.81	27.5	1 029
10	40	2	4	3 000	1.04	30.1	1 075
11	30	3	2	2 000	1.01	24.8	1 017
12	0	2	4	3 000	0.93	30.7	1 071
13	20	2	4	1 000	0.77	18.6	883
14	10	3	6	4 000	0.74	31.8	1 055
15	20	2	4	3 000	0.91	29.8	1 057
16	10	1	2	2 000	0.86	26.3	991
17	10	3	2	4 000	1.09	33.9	1 106
18	30	3	2	4 000	1.11	31.7	1 104
19	20	2	0	3 000	0.98	33.6	1 029
20	10	1	6	2 000	0.80	26.4	1 009
21	10	3	6	2 000	0.95	27.0	1 004
22	20	2	4	3 000	0.86	29.6	1 065
23	20	2	4	5 000	1.15	36.1	1 077
24	10	3	2	2 000	1.09	25.2	1 012
25	20	4	4	3 000	1.03	28.9	1 072
26	30	3	6	4 000	0.77	30.3	1 071
27	10	1	2	4 000	1.09	36.6	1 069
28	30	3	6	2 000	0.87	24.1	9 91
29	20	2	4	3 000	0.97	29.9	1 059
30	30	1	6	2 000	0.69	27.0	980

3.1 Modeling and analysis of variance (ANOVA)

Table 3 shows the ANOVA of the response surface model, coefficient of determination (R^2), adjusted R^2, and predicted R^2 of all the responses models. The differences between adjusted R^2 and predicted R^2 values for each model are all within 0.2, which represents good agreement of the models for the three responses. The adequate precision of the models is greater than 4 as shown in this table, indicating that the models were adequate.

Table 3 Analysis of variance (ANOVA) for the response variables during pelletization of wheat straw

Response variable	Selected model*	R^2	Adjusted R^2	Predicted R^2	Adequate precision
Tensile strength	2FI	0.83	0.78	0.70	16.07
Specific energy consumption	Quadratic	0.95	0.93	0.88	32.01
Pellet density	Quadratic	0.96	0.94	0.86	31.74

Note: * 2FI = 2 factor interaction model; Quadratic = Quadratic model.

Tables 4, 5, and 6 show the analysis of variance (ANOVA) of the response variables (pellet tensile strength, specific energy consumption, and pellet density, respectively) as affected by the binders and interactions after stepwise regression.

Table 4 Analysis of variance (ANOVA) of pellet tensile strength as affected by binder used and compression load

Source [i]	Sum of squares	Degree of freedom	Mean square	F value	P value
Model	0.41	6	0.07	17.76	<0.0001
B	0.02	1	0.02	4.13	0.05
C	0.20	1	0.20	53.30	<0.0001
D	0.07	1	0.07	19.02	0
AD	0.02	1	0.02	4.39	0.05
BD	0.06	1	0.06	15.25	0
CD	0.04	1	0.04	10.47	0
Residual	0.09	23	0		
Lack of fit	0.08	18	0	2.21	0.19
Pure error	0.01	5	0		
Cor total	0.49	29			

Note: [i] A = Wood residue; B = Bentonite; C = Crude glycerol; D = Compression load. The same below.

Table 5 Analysis of variance (ANOVA) of specific energy consumption as affected by binder used and compression load

Source [i]	Sum of squares	Degree of freedom	Mean square	F value	P value
Model	414.73	7	59.25	56.02	<0.0001
A	6.45	1	6.45	6.10	0.02
B	20.59	1	20.59	19.46	0
C	14.14	1	14.14	13.37	0
D	346.56	1	346.56	327.65	<0.0001
CD	6.93	1	6.93	6.55	0.02
D^2	18.14	1	18.14	17.15	0
Residual	23.27	22	1.06		
Lack of fit	21.85	17	1.29	4.53	0.05
Pure error	1.42	5	0.28		
Cor total	438.00	29			

Table 6 Analysis of variance (ANOVA) of pellet density as affected by binder used and compression load

Source [i]	Sum of squares	Degree of freedom	Mean square	F value	P value
Model	54 489.84	9	6 054.43	50.10	<0.0001
B	1 128.57	1	1 128.57	9.34	0.01
C	835.52	1	835.52	6.91	0.02
D	39 787.68	1	39 787.68	329.24	<0.0001
BC	527.69	1	527.69	4.37	0.05
A^2	568.87	1	568.87	4.71	0.04
C^2	1 158.00	1	1 158.00	9.58	0.01
D^2	9 878.65	1	9 878.65	81.74	<0.0001
Residual	2 416.95	20	120.85		
Lack of fit	2 252.91	15	150.19	4.58	0.05
Pure error	164.05	5	32.81		
Cor total	56 906.79	29			

As can be seen from Tables 4, 5, and 6, the probability values indicate that the models are significant. The lack of fit was statistically insignificant as the P values were more than 0.05. An insignificant lack of fit suggests that the model fits the data well. Based on these results, it is indicated that the models sufficiently describe the response surface of pellet tensile strength, specific energy consumption, and pellet density. The response surface model for optimization and prediction was considered reasonable. The final regression model, excluded insignificant terms using the coded factors as independent variables, is expressed by Equations (2), (3) and (4).

$$\text{Tensile strength (MPa)} = 0.94 + 0.026\text{B} - 0.92\text{C} + 0.055\text{D} + 0.032\text{AD} - 0.060\text{BD} - 0.050\text{CD} \quad (2)$$

$$\text{Specific energy consumption (MJ/t)} = 30.27 - 0.52\text{A} - 0.93\text{B} - 0.77\text{C} + 3.80\text{D} - 0.66\text{CD} - 0.79\text{D}^2 \quad (3)$$

$$\text{Pellet density (kg/m}^3) = 1057.72 + 6.86\text{B} - 5.90\text{C} + 40.72\text{D} - 5.74\text{BC} + 4.51\text{A}^2 - 6.43\text{C}^2 - 18.78\text{D}^2 \quad (4)$$

where, A, B, C, and D are the model terms representing wood residue addition (%), bentonite addition (%), crude glycerol addition (%), and compression load (N), respectively.

The predicted vs. experimental values for tensile strength, specific energy consumption, and pellet density are shown in Figures 2a, 2b, 2c showing good agreement between the predicted and actual values. Figures 3a, 3b, 3c show the normal probability plots of the studentized residuals for all the responses. A normal probability plot represents the normal distribution of the residuals, in which case, the points follow a straight line. As shown in Figures 3a, 3b, 3c, it could be concluded that the responses were normally distributed.

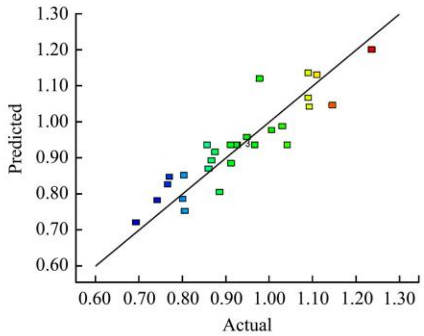


Figure 2a Predicted vs. experimental values of pellet tensile strength (MPa)

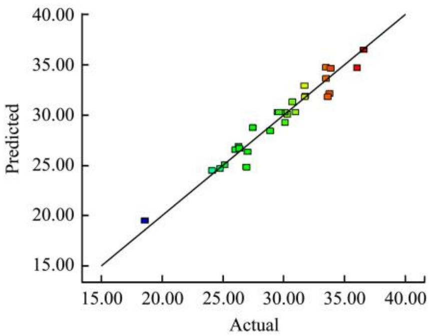


Figure 2b Predicted vs. experimental values of specific energy consumption (MJ t^{-1}) during pelleting

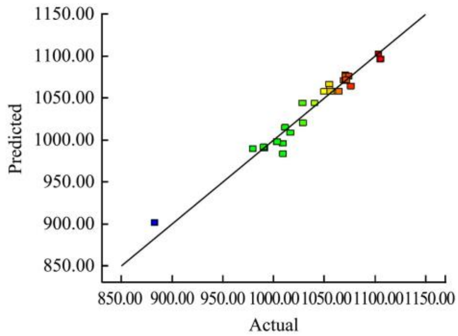


Figure 2c Predicted vs. experimental values of pellet density (kg m^{-3})

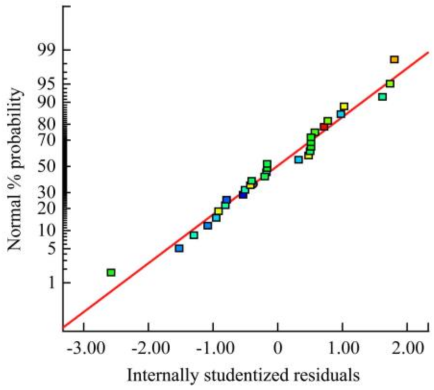


Figure 3a Normal probability plot of the studentized residuals of pellet tensile strength

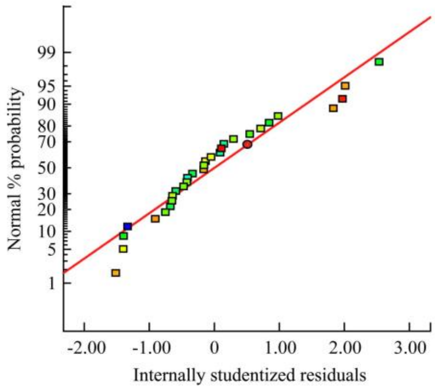


Figure 3b Normal probability plot of the studentized residuals of specific energy consumption

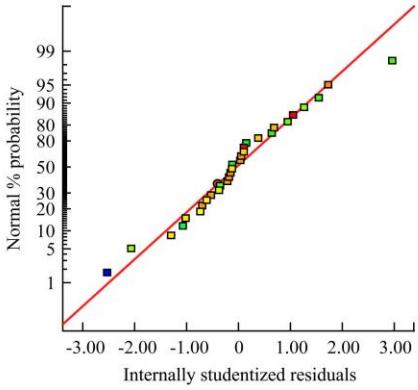


Figure 3c Normal probability plot of the studentized residuals of pellet density

3.2 Tensile strength

The magnitude of the independent variables can be seen from the absolute value of regression coefficients preceding independent variables in Equation (2). The three-dimensional plot of response surfaces of tensile strength as different functions of changes generated by Design Expert software are shown in Figures 4, 5, and 6. In each plot, two independent variables are kept constant while the other two variables can be changed. From Equation (2), Figures 4, 5, and 6, it is evident that compression load had a positive impact while the addition of crude glycerol had a negative effect on tensile strength especially when the compression load was high. It can be seen that crude glycerol was not quite helpful in the bonding of wheat straw particles due to glycerol's liquid characteristic. The interaction effect of wood residue and compression load signifies that the combination of wood residue and compression load has a positive effect on tensile strength. The interaction effect of bentonite and compression load, and crude glycerol and compression load caused a negative effect on tensile strength. As shown in Figure 5, when bentonite was added at 1% compared to 3%, as the compression load increased, the tensile strength increased faster. This indicated that compression load had a bigger effect on tensile strength when smaller amounts of bentonite were added into wheat straw to make pellets. In contrast, compression load will be not that important when the inclusion rate of bentonite is higher.

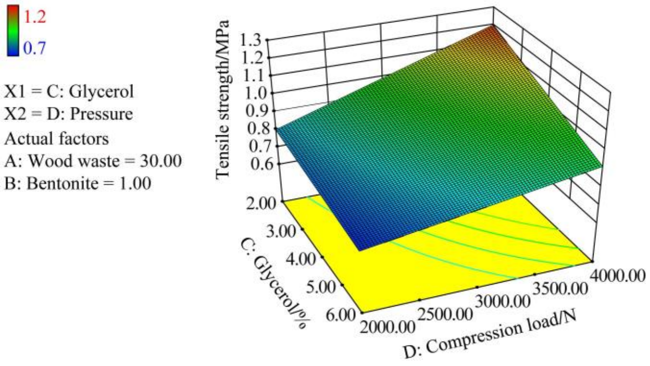


Figure 4 Three-dimensional plot of response surface of pellet tensile strength as function of change in the addition of crude glycerol and compression load (wood residue added at 30% and bentonite added at 1%)

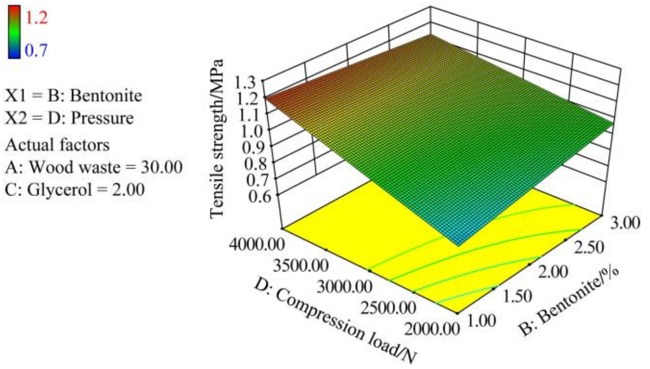


Figure 5 Three-dimensional plot of response surface of pellet tensile strength as function of change in the addition of bentonite and compression load (wood residue added at 30% and crude glycerol added at 2%)

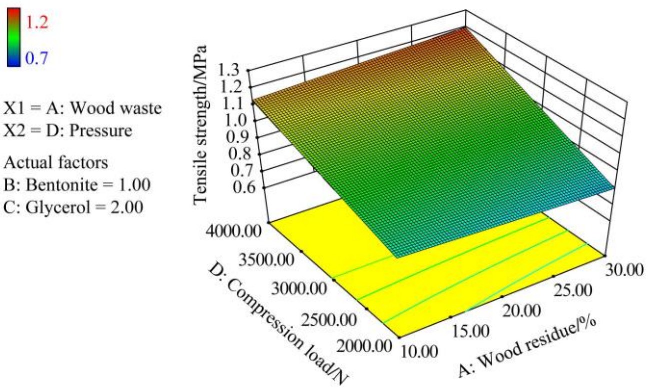


Figure 6 Three-dimensional plot of response surface of pellet tensile strength as function of change in the addition of wood residue and compression load (bentonite added at 1% and crude glycerol added at 2%)

The inclusion rate of wood residue positively affected the tensile strength when the compression load was high, while the inclusion rate of wood residue negatively affected the tensile strength when the compression load was low as shown in Figure 6. It can be concluded that the wood residue particles need a high compression load to achieve small distances between particles so that the natural binders in wood residue would work more effectively to make wheat straw pellets strong.

3.3 Specific energy consumption

The magnitude of the independent variables can be seen from the absolute value of regression coefficients preceding independent variables in Equation (3). The three-dimensional plot of response surfaces of specific energy consumption as different function of changes are shown in Figures 7 and 8.

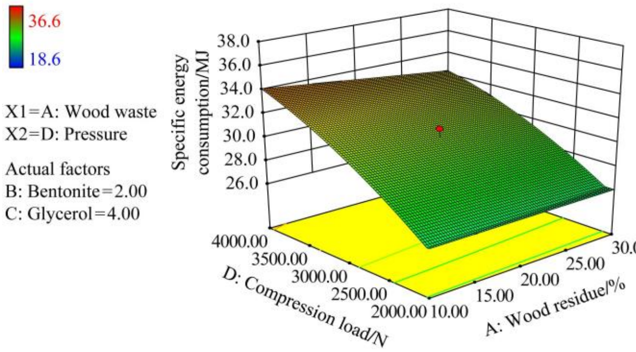


Figure 7 Three-dimensional plot of response surface of specific energy consumption as function of change in the addition of wood residue and compression load (bentonite added at 2% and crude glycerol added at 4%)

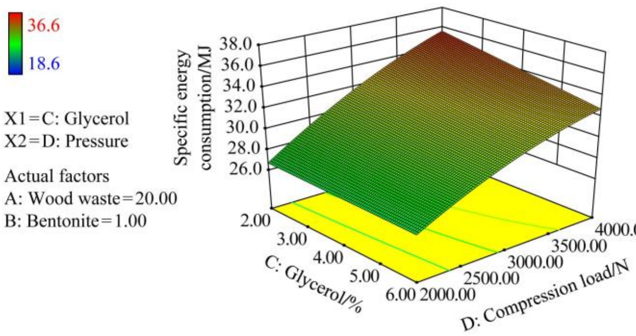


Figure 8 Three-dimensional plot of response surface of specific energy consumption as function of change in the addition of crude glycerol and compression load (bentonite added at 1% and wood residue added at 20%)

From Equation (3), it is obvious that the addition of wood residue, bentonite, and crude glycerol caused the decrease of specific energy consumption. Bentonite and crude glycerol are effective binders for conserving energy as can be seen in Equation (3). It can be concluded that bentonite particles filled in the gaps between wheat straw particles enhancing the binding so that energy could be reserved. Crude glycerol acted as a lubricant when pelleting which resulted in the decrease of the resistance of wheat straw particles. The interaction effect of wood residue and compression load signifies that this combination had a negative effect on specific energy consumption. The interaction effect of crude glycerol and compression load caused negative effects on specific energy consumption as well. The increase of the inclusion rate of wood residue resulted in the decrease of the specific energy consumption when the compression load was high (Figure 7). With the increase of the addition of crude glycerol, specific energy consumption decreased (Figure 8). It is clearly shown in Figures 7 and 8 that specific energy consumption for wheat straw pelleting decreased as the compression load decreased. Compression load was the most significant factor affecting specific energy consumption of pelleting.

3.4 Pellet density

The magnitude of the independent variables can be seen from the absolute value of regression coefficients preceding independent variables in Equation (4). The three-dimensional plot of response surfaces of pellet density as function of different variable changes are shown in Figures 9 and 10. From Equation (4), it can be concluded that compression load positively affected the pellet density while crude glycerol negatively affected pellet density. The interaction effect of bentonite and crude glycerol resulted in negative effect on pellet density.

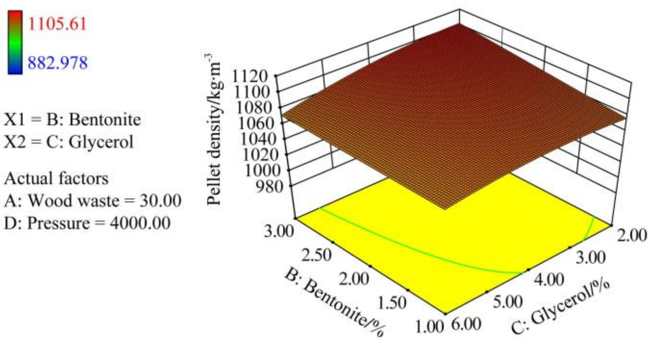


Figure 9 Three-dimensional plot of response surface of pellet density as function of change in the addition of crude glycerol bentonite (wood residue added at 30% and compression load at 4 000 N)

It is shown in Figure 9 that pellet density increased with the increase of the bentonite inclusion rate from 1% to 3% when the addition of crude glycerol and the compression load was at 2% and 4 000 N, respectively.

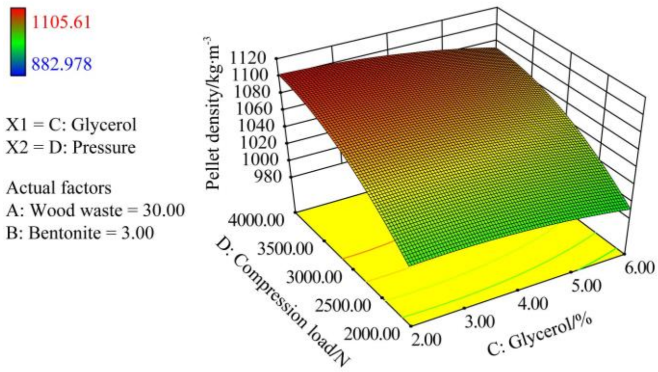


Figure 10 Three-dimensional plot of response surface of pellet density as function of change in the addition of crude glycerol and compression load (bentonite added at 3% and wood residue added at 30%)

Pellet density decreased with the increase of the inclusion rate of crude glycerol especially when the compression load is high (4 000 N) (Figure 10). If the inclusion rate of crude glycerol increased, the pellet density and pellet tensile strength would decrease but energy for pelleting would increase.

3.5 Optimization and model validation

3.5.1 Goals of optimization

The goals of the optimization are shown in Table 7. High tensile strength, low energy consumption, and high pellet density were the goals for responses. For the goals related to variables, the addition of wood residue and crude glycerol was in range while the bentonite inclusion rate was minimized due to its high ash content. The goal of compression load was considered to be maximized as the compression load is usually high in the real pelleting process.

Table 7 Goal for optimization of variables during the experimental pelletization of wheat straw

Name	Goal
Wood residue (%)	In range (0 to 30)
Bentonite (%)	Minimize (0 to 3)
Crude glycerol (%)	In range (0 to 6)
Compressive load (N)	Maximize
Tensile strength (MPa)	Maximize
Specific energy consumption (MJ/t)	Minimize
Pellet density (kg/m^3)	Maximize

3.5.2 Other goals

Heating value, ash content, and cost were also considered to be minor factors that affect the final optimization. The heating value, ash content and cost of the binders are shown in Table 8. Crude glycerol had the highest heating value among all binders. Wood residue had a higher heating value compared to wheat straw. There was no heating value enhancement by adding bentonite. In terms of ash content, wood residue and crude glycerol contained less ash than wheat straw. Bentonite has very high ash content. However, the cost of bentonite is the lowest among all binders considered. So the inclusion rate of bentonite should not be too high.

Table 8 Heating value, ash content, and cost of binders and wheat straw

Factor	Wheat straw	Wood residue	Bentonite	Crude glycerol
Heating value/MJ t^{-1})	17.74	20.13	0.00	27.10
Ash content/%	7.88	0.47	92.90	2.00
Cost /CAD \$ t^{-1}	40	150	75	110

3.5.3 Final optimization and model validation

A number of optimization options results were given by design expert and presented in Table 9. In Table 9, the combinations with ash content above 10% were excluded. Option 18, wheat straw pellets with 30% wood residue, 0.80% bentonite, 3.42% crude glycerol addition and 4 000 N of compressive load, was chosen to be the best one due to the low ash content of 6.13%, high heating value of 18.64 MJ/t with a total material cost of CAD$ 60.70 per ton.

Conformity tests were conducted with the optimum experimental conditions to validate the adequacy of the models. The experimental result of the tensile strength, specific energy consumption, and pellet density values of the optimization option were 1.14 MPa, 32.6 MJ/t, and 1 094 kg/m^3, respectively. The percentage error was calculated as:

Percent Error (PE)=|observed values−predicted values|/ predicted values ×100%

The calculated PE for tensile strength, specific energy consumption and pellet density were 2.70%, 4.07%, and 1.34%, respectively. It is obvious that the models were able to predict the response variables with acceptable accuracy.

Table 9 Final optimization options and their responses (options with ash content >10% were excluded).

Option	Wheat straw/%	Wood residues/%	Bentonite /%	Crude glycerol/%	Compression load/N	Heating value/ MJ kg^{-1}	Ash content/%	Cost /CAD \$*	Tensile strength/MPa	Specific energy consumption/MJ t^{-1}	Pellet density /kg m^{-3}
1	66.58	30.0	2.25	1.17	3 301	18.17	7.49	59.67	1.12	31.9	1 074
2	66.57	30.0	2.25	1.18	3 297	18.17	7.49	59.68	1.11	31.8	1 074
3	66.59	30.0	2.25	1.16	3 300	18.17	7.49	59.67	1.12	31.9	1 074
4	66.57	30.0	2.26	1.17	3 294	18.17	7.50	59.68	1.12	31.8	1 074
5	66.62	30.0	2.26	1.12	3 267	18.16	7.50	59.64	1.11	31.7	1 073
6	66.64	30.0	2.24	1.12	3 316	18.16	7.48	59.64	1.12	31.9	1 075
7	66.62	30.0	2.28	1.10	3 293	18.16	7.52	59.64	1.12	31.8	1 074
8	66.60	30.0	2.27	1.13	3 250	18.16	7.51	59.65	1.11	31.7	1 072
9	66.68	30.0	2.19	1.13	3 308	18.17	7.44	59.62	1.12	32.0	1 073
10	66.59	30.0	2.31	1.10	3 269	18.15	7.54	59.65	1.12	31.7	1 074
11	66.68	29.9	2.24	1.16	3 291	18.17	7.49	59.62	1.11	31.8	1 074
12	66.68	29.9	2.24	1.17	3 300	18.17	7.49	59.62	1.12	31.9	1 074
13	66.45	30.0	2.26	1.29	3 272	18.18	7.49	59.76	1.11	31.7	1 074
14	66.48	30.0	2.36	1.16	3 311	18.15	7.58	59.71	1.12	31.8	1 076
15	66.35	30.0	2.29	1.36	3 228	18.18	7.51	59.82	1.10	31.4	1 073
16	66.60	30.0	2.08	1.32	3 446	18.21	7.34	59.71	1.13	32.5	1 077
17	65.58	30.0	2.36	2.06	3 947	18.23	7.53	60.34	1.14	33.3	1 094
18	65.78	30.0	0.80	3.42	4 000	18.64	6.13	60.70	1.11	33.9	1 079

Note: * cost (CAD$) of materials for pelleting (wheat straw and binders).

4 Conclusions

The central composite rotatable design technique with four-factor, five-level full-factorial design matrix was used to predict tensile strength, specific energy consumption, and pellet density of wheat straw pellets. The models were derived and subjected to ANOVA and diagnostic analysis. The values of adjusted R^2 of the model for tensile strength, specific energy consumption, and pellet density were 0.78, 0.93, and 0.94, respectively. The effect of the addition of wood residue, bentonite, crude glycerol, and compressive load on pellet tensile strength, specific energy consumption during pelleting, and pellet density was indentified and discussed. Optimization considering both responses and other factors (heating value, ash content, and cost of binders) was conducted. The optimal combination for making wheat straw pellets was wheat straw with the addition of 30% wood residue, 0.80% bentonite, 3.42% crude glycerol and 4 000 N of compressive force, in which case the tensile strength, specific energy consumption, pellet density values, ash content, and heating values were 1.14 MPa, 32.6 MJ/t, 1 094 kg/m^3, 6.13%, and 18.64 MJ/t, respectively. Confirmation tests were carried out and the results indicated that the model was highly accurate. Pilot-scale experiments need to be done in the future to verify and achieve the final optimization combination of the binders.

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