Effect of Caragana and Corn Straw Mixture Parameters on Pellet Feed Unit Density

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The unit density value is a key quality index for pellet feed production. This study presents an experimental evaluation of the unit density for the pelletizing of caragana and corn straw, under different levels of technological parameters, including moisture content, weight ratio of caragana, and particle size. Results showed that these three parameters of raw materials affected unit density. Through orthogonal test and extreme variance analysis, it was shown that the various moisture content and weight ratio of caragana had a significant effect on the density of pellets, and the influencing factors were ranked as moisture content > weight ratio of caragana > particle size of the materials. In compliance with industry standards, optimizations of the parameters resulted in a granulation density of 1.15 g/cm^3 with particle size of 5 mm, moisture content of 13.4% and weight ratio of caragana of 24.8%.

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INTRODUCTION

Caragana is a forage that is high in protein, balanced in amino acids, and has high overall nutritional value, while corn straw is rich in organic matter, high in fibre, and palatable (Nie *et al*. 2023). Both are usually used as mixed feedstuffs for ruminants (Zhang *et al*. 2010). Corn straw and caragana are crushed or kneaded, shredded, and then processed into pellets using a granulator, which is the typical form of feed utilization at present. Corn straw and caragana mixed pellet feed can make efficient, low-cost use of local livestock resources, while at the same time their anti-inflammatory, antioxidant, crude fibre, crude fat digestibility and weight gain are all better than feeding corn straw or caragana separately (Lv *et al*. 2021). Research data has demonstrated that the caragana-straw mixture can better meet the requirements of cows to eat effective fibre, resulting in higher milk fat, milk protein, and other nutrients (Ma *et al*. 2020). Pellet feed unit density is an important indicator in feed production (Chen *et al*. 2018), reflecting the palatability and nutrient density of pellet feeds (Wang *et al*. 2018). Low unit density pellets are friable. High unit density triggers increased hardness and poor palatability of the pellets. At the same time, unit density affects the storage of pellet feed (Abdollahi *et al*. 2019); therefore, the unit density value is a key quality evaluation index for pellet feed production.

There are many examples of mixing corn straw with concentrated feeding stuff, fodder mulberry, alfalfa, *etc.* to make pellet feed for ruminants (Lu *et al*. 2020; Du *et al*. 2021). When mixing forage with concentrated feeding stuff for pelleting, the results showed that an increase in the weight ratio of forage decreased the unit density of the pellets, while an increase in the weight ratio of alfalfa resulted in increase in unit density (Ge *et al*. 2021). While studying the effect of different ratios of caragana mixed fermented feeds on the moulding quality of pelleted feeds, it was similarly found that the unit density of pellets decreased significantly as the weight ratio of caragana increased (Yang *et al*. 2023). When studying the performance of caragana and alfalfa mixed pelleting, it was found that the unit density of the pellets first increased as the ratio of caragana increased, and then it gradually decreased beyond 35% (Chao *et al*. 2009). It was found that the unit density varied in weight ratio of the forage mixes after pelleted and that the nutrient content changed (Shi *et al*. 2022).

Forage dense moulding is mainly based on mechanical inlay, with solid bridging formed by natural binders (cellulose, crude protein) Kaliyan and Morey (2010). The moisture content (Ma *et al*. 2022), particle size (Kong *et al*. 2019), and weight ratio of forage (Zhang *et al*. 2018) all affect the pelleting temperature of the moulding die holes, resulting in the deformation of the crude protein and the lignin glassy state is one of the key factors affecting the density of the pellet feed (Anukam *et al*. 2019). The above factors also affect the mechanical inlay effect. Pelleting pressure, temperature, degree of forage crushing, and moisture content all affect the density of forage pellets (Mani *et al.* 2006). The interaction between various parameters in the dense moulding process of corn straw, temperature, raw material moisture content, and pressure of rollers will have a significant effect on the unit density of pellets (Ren *et al*. 2012; Wang *et al.* 2016). Barley straw particles with a moisture content of 19% to 23% achieves the best pelleting effect, with a unit density close to 1.0 g/cm³ (Serrano *et al.* 2001). In the process of alfalfa dense moulding, the effect of moisture content on the pressure of hay moulding was very significant, and the pressure on hay was exponentially related to the density of pellets (Ma *et al.* 2022). The raw material characteristics affect the quality of olive branch pellets, and the results showed that the factors affecting the density and durability of the pellets were, in descending order, temperature, moisture content, particle size, and extrusion pressure (Caronen *et al.* 2011). The experiment found that higher forage fibre significantly increased the unit density of pelleted feeds (Li *et al.* 2022). For caragana, particle size, moisture content, and high temperature (in that order) are important factors for increasing unit density and durability (Zhang *et al.* 2014), with pressure as a marginal influence (Han *et al.* 2014). Forage particle size affects dense moulding density, the smaller the particle size of the raw material, the easier it is to produce deformation bonding (Wang *et al.* 2021). However, the relative density does not always increase as the particle size decreases (Wang *et al.* 2021).

The main objective of this work was to study the effect of pellet feed pelleting of caragana and corn straw to explore the utilization of pasture resources in Inner Mongolia and to serve the animal husbandry industry. In this study, the effect of factors on pellet unit density of mixed raw material (caragana and corn straw) were investigated. Subsequently, the relationship between the factors and unit density was analyzed using response surface method to obtain the effect of the interaction of various influencing factors on the pellet unit density, and the regression equation of the effect of various factors on the unit density.

EXPERIMENTAL

Materials and Methods

Caragana plants were selected and cut in June 2022 from Ulanqab City, Hohhot city, Inner Mongolia Autonomous Region, China. The caragana plants used were 3 years old. Corn straws were selected in June 2022 from Tumd Right Banner, Hohhot city, Inner Mongolia Autonomous Region, China after the corn harvest season. The single factor test was used to determine that the moisture content (MC), weight ratio of caragana (WRC), and particle size (PS) would be the factors used on unit density testing.

According to the general technical specification of ring die extruder national standard GB/T 20192 (2006), the MC of the material needs to be controlled. Through literature survey, the MC of raw materials for better dense moulding of corn straw and caraganan is around 17% (Wang *et al.* 2016) and 15% to 18% (Yang *et al.* 2023). By single factor test, the MC of mixed raw material for testing was adjusted into 10%, 15%, and 20%. The method used for adjusting MC was calculated as follows,

$$m_1 = \frac{m_w}{1 - m_w} \times m_2 \tag{1}$$

where m_w is the MC (%), and m_1 is water addition, and m_2 is the weight of material (g) after drying.

The quality testing and grading of natural forage pellets enterprise standard Q/NCFY 002 (2020) was followed. The crude protein and acid detergent fiber in corn straw and caragana determines its ability to make only second class feeding stuff with less than 30% caragana. A single factor test was used to determine the WRC value.

Raw material particle size affects granulation and nutrient absorption of pellet feed. Many researchers have set the particle size of forage below 3 mm (Kong *et al*. 2019; Lv *et al*. 2016), but in actual production due to cost considerations it is usually to set the particle size above 5 mm. A single factor test was used to determine the PS value.

Raw materials were crushed by means of a hammer mill (550-type, Yuying Co., Ltd., Sichuan, China) and sieved using screens of mesh sizes of 3, 4, and 5 mm separately. Based on the nutritional requirements of crude protein (Ming *et al.* 2018), the factor levels of raw material were set as shown in Table 1.

Table 1. Test Factor Levels of Quadratic Response Surface

Variable Levels	A: PS (mm)	B: MC (%)	C: WRC (%)
-1	3	10	20
0	4	15	25
1	5	20	30

Pelletizing Process

The methodology and pellet extruder used in this study are illustrated in Fig. 1 (self-designed and made). The pelletizing process was carried out with a ring die extruder that has a barrel diameter of 6 mm, with a length to diameter (L/D) ratio of 4.75:1 (De *et al*. 2020), with press roll speed of 60 r/min. Every 5 min, the temperature measurements were taken on the ring die inner and outer walls. When there were three consecutive temperature differences of less than 5 °C, the process was considered to have reached a steady state.

The pellets were allowed to cool and dry for approximately 24 h under ambient conditions of room temperature (approximately 20 ◦C) to equilibrate the final moisture to a level between 8 and 10% and stored in sealed polyurethane bags at 4 °C for analysis.

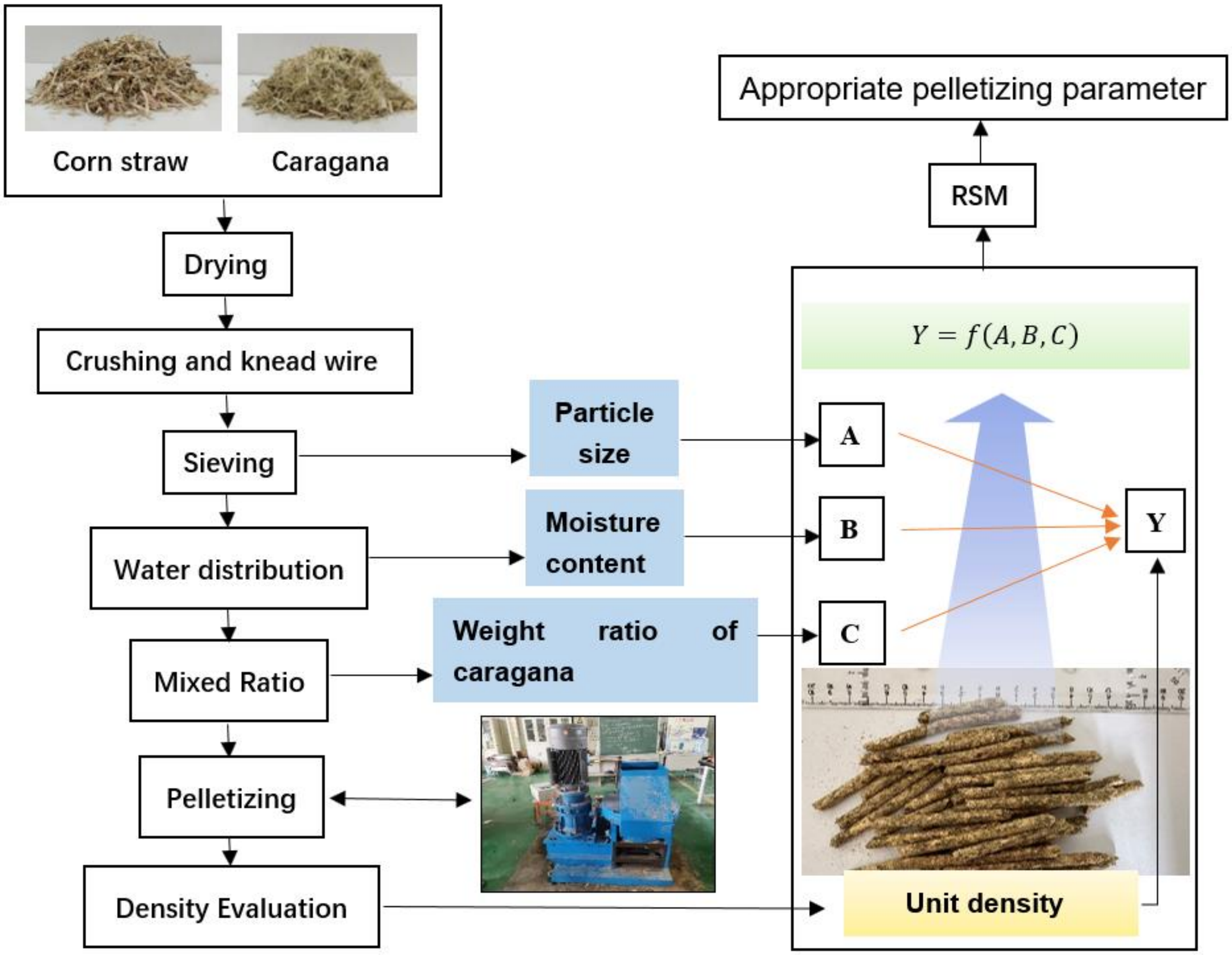

Fig. 1. Methodology pelletizing

Determination of Unit density of Pellets

According to Eq. 2, the unit density was calculated by weighing the pellet and measuring its dimensions using a Vernier caliper. The length of each pellet was about 30 mm. The ends of the pellets were uneven and needed to be smoothed out with 0# abrasive paper. 100 samples were taken from each group and averaged. The density was calculated as follows,

$$\rho = \frac{m}{\pi\left(\frac{d}{2}\right)^2 l} \tag{2}$$

where m is the weight of pellet (g), l is the length of pellet (cm), ρ is the unit density of pellet (g/cm^3), and d is the diameter of the pellet (cm).

RESULTS AND DISCUSSION

The experimental results from pelleting of caragana and corn straw mixture based on the quadratic response surface method are shown in Table 2. The value of unit density response values varied over a range within the experimental scheme, thus it can be seen that each of the selected factors had a corresponding effect on the pellet feed forming unit density.

Table 2. Experimental Design Matrix and Response of Experiments Used for Unit Density of Pellets

Run	A: PS (mm)	B: MC (%)	C: WRC (%)	Y: Unit Density (g/cm^3)
1	4	15	25	1.16
2	4	20	20	1.09
3	4	15	25	1.13
4	4	20	30	1.08
5	4	15	25	1.12
6	3	15	30	1.13
7	5	15	20	1.13
8	4	10	20	1.04
9	5	20	25	1.08
10	4	10	30	1.10
11	4	15	25	1.16
12	3	10	25	1.10
13	5	15	30	1.11
14	4	15	25	1.13
15	3	15	20	1.08
16	3	20	25	1.12
17	5	10	25	1.14

The data were analyzed using Design-Expert 12.0 (Stat-Ease, Minneapolis, MN, USA) to obtain a polynomial regression equation with pellet feed unit density as the objective response function. The equation with the three test factors of WRC, MC, and PS as dependent variables is shown in Eq. 3:

$$Y_1 = 1.13 + 0.0137\text{c}A + 0.0137B + 0.0188C - 0.01AB - 0.0175AC - 0.0088BC + 0.00256A^2 - 0.0081B^2 - 0.03C^2 \quad (3)$$

where Y_1 is the unit density of pellet (g/cm^3), A is particle size (mm), B is moisture content (%), and C is weight ratio of caragana (%).

Table 3 shows the ANOVA of the factors affecting the pellet unit density. The table shows that the F-value of the model described in this test was 4.89 and the P-value was 0.0241. The fact that this value was less than 0.05 means that the model of the test data was significant. The fitting coefficient of determination R^2 of the regression equation was 0.8628, which indicates that the model was well fitted and was able to describe the relationship between each influence factor and the response value.

Through analyzing the regression equation for pelleting unit density, it can be concluded that the coefficients of the primary terms of each factor as well as the coefficients of the interaction terms were independent of each other in this data model. Therefore, the comparison of the magnitude of the absolute values of the coefficients of the primary terms of the different factors can be used to determine the effective magnitude of the different factors on the pelleting unit density of the caragana and corn straw mixture.

The ANOVA shows that the various values of MC and WRC had a significant effect on the density of pellet, and the influencing factors were ranked as MC > WRC > PS of the materials.

Table 3. ANOVA Results of Dependent Variables on Pellet Unit Density (g/cm^3)

Source	Sum of Squares	df	Mean Square	*F*-value	*P*-value
Model	0.0137	9	0.0015	4.89	0.0241
A-PS	0.0010	1	0.0010	3.25	0.1146
B-MC	0.0027	1	0.0027	8.67	0.0216
C-WRC	0.0019	1	0.0019	6.03	0.0437
AB	0.0016	1	0.0016	5.15	0.0575
AC	0.0012	1	0.0012	3.94	0.0875
BC	0.0012	1	0.0012	3.94	0.0875
A^2	0.0000	1	0.0000	0.0847	0.7795
B^2	0.0044	1	0.0044	14.31	0.0069
C^2	0.0038	1	0.0038	12.20	0.0101
Lack of Fit	0.0008	3	0.0003	0.7381	0.5819
Pure Error	0.0014	4	0.0003		
Cor Total	0.0158	16			

Effect of Moisture Content on Unit Density

In the forage dense forming process, within a certain range of changes in moisture content, can ensure the adhesion between the raw materials. This is because the water molecules in a certain temperature condition can promote the adhesion between the dry raw material inside the paste *via* the friction effect caused by the temperature inside the pelleting ring mold (Huang *et al*. 2017). This results in the increase of the unit density of the pellets. To investigate the effect of water content on the unit density of pellets, the experimental parameters are designed as PS of 5 mm, WRC of 25%, and MC adjusted to 5%, 10%, 15%, 20%, and 25% respectively. The results are shown in Fig. 2.

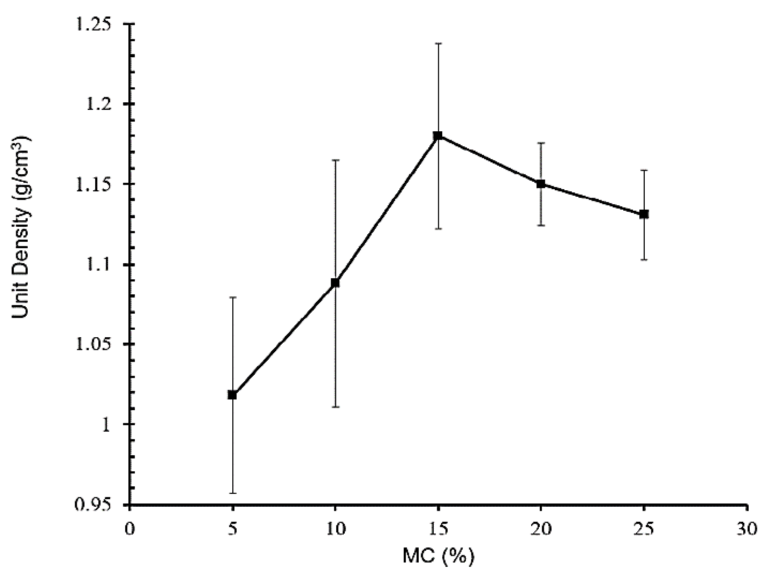

Fig. 2. The effect of MC on pellet unit density

It can be seen from Fig. 2 that at first the unit density increased with increasing water content. The unit density of the pellets tended to decrease when the MC of the raw material reached 15%, when the unit density of the pellets was at 1.18 g/cm^3. The unit density of the pellets was at its peak in the interval set for the test, and it decreased to a lesser extent as the MC of the material increased further.

The friction between the raw materials, especially in the die holes, increased the temperature, which was measured as being up to 140 °C. High temperature is an important factor in the dense moulding of forage. When the moisture content of the raw material increases, it leads to the generation of a higher amount of water vapor. During pelleting, vapor reduces the friction coefficient between the pellet surface and the forming die holes, and the extrusion time of the pellets is shortened. The materials that have not reached the dense state are extruded, thus making the extrusion pressure in the center of the pellets greater than that at the edges. This leads to a layered fracture of the pellets along the outer surface. This condition of the pellet surface is shown in Fig. 3 and it reduces the unit density of the particles.

As the temperature rises, the water within the forage vaporises and its content in the pellets decreases, resulting in poor heat transfer due to large distances between particles. With the increase of MC, the distance between particles increases trapping water in void, and the excessive internal pressure from vapor is generated inside the pellets. The intermolecular forces (hydrogen bonds and Van der Waals forces) are not able to form, due to spaces between the particles. Such effects weaken the binding forces between particles (Zafari and Kianmehr 2012), and the unit density of the pellets decreases. During pelleting, vapor reduces the friction coefficient between the pellet surface and the forming die holes, and the extrusion time of the pellets is thereby shortened.

Fig. 3. Surface cracks of pellets

Effect of Weight Ratio of Caragana on Unit Density

Crude protein and crude fibre contents in caragana play an important role in the unit density of pellet feed. The protein denaturation and lignin glass transition were triggered at high temperature due to friction, which further increased inter-particle adhesion and enhanced pellet feed unit density (Thomas and van der Poel 2020). Therefore, an increase in caragana weight can increase pellet unit density. However, the experimental parameters were designed as PS of 5 mm, MC of 15%, and adjusted WRC as 15%, 20%, 25%, 30% and 35%. The peak unit density of pellet was 1.22 g/cm^3 at 20% of WRC, not at the maximum WRC content of 35%. The results are shown in Fig. 4. This may be because the fibrous structure of caragana tends to crowd the space during the pelleting. The low caragana content leads to a decrease in kneaded material, while powdered caragana and corn straw granules are involved in pelleting. The increased number of powder particles in contact enhances the molecular attraction (Tian *et al*. 2011), including electrostatic attraction (Calmes and O'Brien 2017). The powder particles fill the space of kneading material, forming a solid inter-particle filling, and embedding (Kaliyan and Morey 2010), which ultimately increases the unit density.

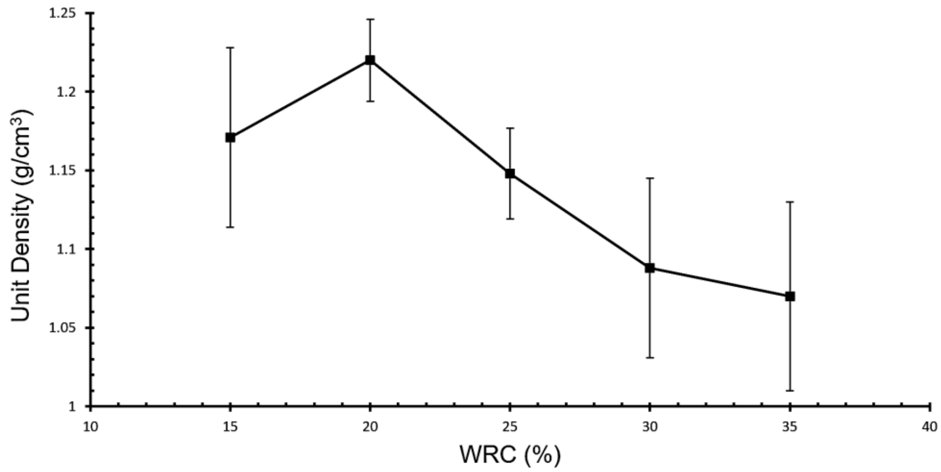

Fig. 4. The effect of WRC on pellet unit density

Effect of Factor Interactions on Unit Density

The interaction between the test factors can be observed by RSM. Figure 5(a) reflects the interactive effect of two factors, WRC and PS, on unit density of pellet. As the level of raw material PS gradually increased, the unit density of pellet increased gradually, and then it levelled off as the percentage of caragana increased, and the data showed a saddle-shaped surface, with the values changing more rapidly at lower levels for both factors. After crushing, caragana are in the form of kneaded threads, unlike corn straw which are clumped together into flocs. Flocculent raw materials do not easily enter the die holes, resulting in moulding difficulties (Chao *et al*. 2009). With an increase in PS value and an increase in caragana, the flocs will be longer, more numerous, and more prone to clumping, resulting in reduction of pellet unit density.

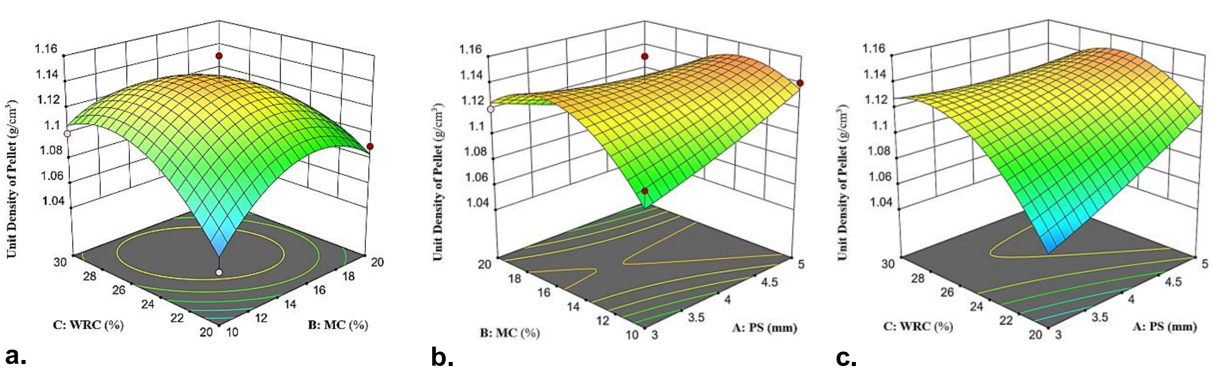

Fig. 5. Response surface plots of pellet unit density for effects of MC, WRC, and PS

Figure 5(b) reflects the interaction of PS and MC on the density of granulation. As the PS of the material gradually increased, the effect of MC on the unit density tended to increase and then gradually decrease. This may because that the caragana are kneaded, unlike corn straw, which clumps together into flocs. In the case of small PS, the friction is insufficient and the unit density is low. In the case of big PS, the single particle itself has relatively low density, and the deformation is relatively small, so the unit density of the pellet is small. When PS value was in the range 4 to 5 mm, the unit density reached approximately to the maximum limit.

Figure 5(c) reflects the interaction effect of MC and WRC on unit density. Increases in WRC and MC within the corresponding levels increased and then decreased for unit density, and the value of unit density was higher at intermediate levels of both factors, which the surface diagram presents spherical-type geometry.

According to the industry standard SC/T 6020 (2002) of the Ministry of Agriculture of the People's Republic of China, the unit density of straw pellet ranges from 0.9 to 1.2 g/cm^3 without steam modulation. Therefore, the results of this test are in accordance with the requirements; combined with the quadratic regression equation for optimization, to explore the better combination of factors at the corresponding level interval. Based on the level and unit density data of the factors in the experiment, the optimization data setup is shown in Table 4.

Table 4. Parameter Optimization Setting Table

Test Factors	Optimization Targets	Min Value	Max Value
PS (mm)	3 ≤ x ≤ 5	3	5
MC (%)	10 ≤ x ≤ 20	10	20
WRC (%)	20 ≤ x ≤ 30	20	30
Unit Density (g/cm^3)	x ≤ 1.17	1.04	1.16

Optimization for the pellet unit density was carried out, in which 35 groups met the requirements. Among them, the groups of solutions with higher expectation values are shown in Table 5.

Table 5. Parameter Optimization Results

Run	PS (mm)	MC (%)	WRC (%)	Unit Density (g/cm^3)	Expected values
1	5.00	13.41	24.84	1.15	0.915
2	5.00	13.39	24.85	1.15	0.915
3	5.00	13.43	24.83	1.15	0.915
4	5.00	13.39	24.83	1.15	0.915
5	5.00	13.32	24.88	1.15	0.915
6	5.00	13.27	24.81	1.15	0.914
7	5.00	13.71	24.79	1.15	0.914
8	5.00	13.73	24.81	1.15	0.914
9	5.00	13.61	24.51	1.15	0.913
10	5.00	13.78	24.78	1.15	0.913

The combination with the highest value of pellet unit density in the preset range were PS (5 mm), MC (13.4%), WRC (24.8%), and the pellet density under this condition was 1.15 g/cm^3.The result is consistent with those of Ma *et al*. (2021) and Huang *et al*. (2017), and the unit density met the requirements of the industry standard.

CONCLUSIONS

1. Caragana and corn straw can be mixed into a pellet feed. The resulting pellets obtained reasonable unit density values that can meet the requirements of the industry standard.
2. For the pelletizing of caragana and corn straw mixtures, the effective magnitude of each test factor on unit density within the corresponding level were MC, WRC, and PS.
3. Optimization of the test parameters resulted in pellet unit density of 1.15 g/cm^3 with PS of 5 mm, MC of 13.4%, and WRC of 24.8%. The optimal process parameters are only valid for the specific equipment that has been used in this experiment.
4. A mixture of caragana and corn straw can be formed into a feed pellet that can have reasonable unit density values that can meet the industry standard.
5. For the pelletizing of caragana and corn straw mixtures, the effective magnitude of each test factor on unit density within the corresponding level were moisture content (MC), weight ratio of caragana (WRC), and particle size (PS).
6. Optimization of the test parameters resulted in pellet unit density of 1.15 g/cm^3 with PS of 5 mm, MC of 13.4%, and WRC of 24.8%. The optimal process parameters are only valid for the specific equipment that has been used in this experiment.

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