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Research Paper

Effect of process variables on the quality characteristics of pelleted wheat distiller's dried grains with solubles☆

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The rapid expansion of ethanol processing plants in Canada has resulted in a significant increase in the production of wheat-based distiller's dried grains with solubles (DDGS). Transportation and flowability problems associated with DDGS necessitate investigations on pelleting. In the present study, the effect of process variables like die temperature (T) and feed moisture content (M_w) on the pellet properties like pellet moisture content, durability and pellet density was explored using a single pelleting machine; further studies on pelleting DDGS using a pilot-scale pellet mill were also conducted to understand the effect of die diameter and steam conditioning on durability and bulk density of pellets. Proximate analysis of DDGS indicated that crude protein and dry matter were in the range of 37.37–40.33% and 91.27–92.60%, respectively. Linear regression models developed for pellet quality attributes like pellet moisture content, pellet density and durability adequately described the single pelleting process with R^2 value of 0.97, 0.99 and 0.7, respectively. ANOVA results have indicated that linear terms T and M_w and the interaction term $T \times M_w$ were statistically significant at $P < 0.01$ and $P < 0.1$ for pellet moisture content and pellet density. Based on the trends of the surface plots, a medium T of about 50–80 °C and a low M_w of about 5.1% resulted in maximum pellet density and durability and minimum pellet moisture content. Results from pilot-scale studies indicated that bulk density, durability and throughput values were 436.8–528.9 kg m^{-3}, 60.3–92.7% and 45.52–68.77 kg h^{-1}, respectively. It was observed that both die diameter and steam addition had a significant effect on the bulk density and the durability values. The highest bulk density and durability were achieved with 6.4 mm die diameter with steam addition compared to 7.9 mm die with or without steam addition.

1. Introduction

The production of biofuels around the world is increasing significantly with the aim of reducing greenhouse gas emissions, preventing climate change and also reducing dependence on petroleum. The Canadian government has laid out plans to increase biofuels production. The government is providing assistance to producers and the private sector in the

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form of loans and subsidies to companies to produce biofuels, to create new jobs and to strengthen rural economies. This has in turn resulted in rapid expansion of the fuel ethanol industry. It was projected that by 2007, the production of fuel ethanol in Canada would be one billion litres per year and the production capacity is expected to increase to two billion litres by 2010 to meet the Canadian federal renewable fuel standard (Canadian Renewable Fuels Association, 2009).

Distiller's dried grains with solubles (DDGS) is a co-product from the fermentation of cereal grains to produce ethanol and carbon dioxide in biofuel and beverage ethanol industries. The feedstock of choice for the production of fuel ethanol for biofuel in Western Canada is wheat. Currently, Western Canada has seven fuel ethanol processing plants with an annual fuel ethanol production of 502 million litres (Canadian Renewable Fuels Association, 2009). The large-scale production of ethanol biofuel in Western Canada has resulted in availability of high volumes of the wheat-based DDGS. Earlier, DDGS was considered as a by-product, with little or no value and was sold locally to feed mills as they are produced in moderate quantities. At present, DDGS is used widely in feed rations as an inexpensive source of protein and energy, often as substitutes for corn and soybean meal, both of which have increased in price because of the demand for corn by the ethanol industry. A number of researchers have worked on incorporation of DDGS in poultry and aquafeed diets (Chevanan, Muthukumarappan, & Rosentrater, 2007a, 2007b; Chevanan, Rosentrater, & Muthukumarappan, 2007; Chevanan, Rosentrater, & Muthukumarappan, 2008a, 2008b; Kannadhason, Muthukumarappan, & Rosentrater, 2009).

DDGS is a low density material (380–440 kg m^{-3}) and also has flowability problems during loading and unloading into storage bins (Rosentrater, 2006a, 2006b). DDGS in granular form is loaded into trucks and transported from ethanol production facilities. Increase in the cost of conventional fuels has resulted in significant increase in the cost of transporting the DDGS in granular form. Schlicher (2005) reported that caking might limit the flowability of granular DDGS in railcars, resulting in shippers being required to pay extra for shipping. Rosentrater (2006b) indicated that caking of the granular DDGS poses a challenge during the unloading of the product which might require extra machinery, labour, expenses to unload, and railcar downtime.

Improving the bulk density of DDGS by densification can help to overcome the limitations associated with storage, transportation, cost of shipping and handling. Densification using either a pellet mill or an extruder can be a viable and promising solution to increase the density of DDGS. Pelleting of DDGS using a pellet mill or an extruder offers many advantages like versatility, high productivity and quality, possibility of product design, absence of effluents during processing, and improved functional characteristics of protein source without losing protein quality (Harper, 1981; Shankar, Sokhansanj, Bandyopadhyay, & Bawa, 2008).

Rosentrater (2007) has worked on pelleting 100% corn-based DDGS using a commercial feed pellet mill without incorporating any binding agent. He reported that the heating used in the pelleting process did not harm the high-protein and low-starch nutrient content of the pelleted DDGS. The Agricultural Utilization Research Institute and Minnesota Corn Growers Association (AURIMCGA, 2005) conducted pelleting studies on DDGS with moisture content, pellet die and steam addition as pelleting variables. It was reported that higher die compression ratio and steam addition increased the durability and bulk density of the DDGS pellets. Chevanan et al. (2008a, 2008b) extruded ingredient blends containing 20%, 30% and 40% DDGS. They indicated that increasing the DDGS content from 20% to 40% resulted in a 37.1%, 3.1%, and 8.4% decrease in the extrudate durability, specific gravity and porosity, respectively, however the bulk density increased by 7.5%. Much of literature available on pelleting and extrusion studies is on corn-based DDGS with emphasis on the nutritional aspects. Not much literature is available on how the process variables like die temperature, feed moisture content, die diameter and steam conditioning affect the quality of pellets from wheat-based DDGS.

The aim of the present research is to study the effect of the pelleting process variables on the quality attributes of wheat-based DDGS pellets. The specific objectives are to study the pellet characteristics during single pelleting, develop regression models for the quality attributes like pellet density, moisture content and durability in terms of die temperature and feed moisture content, and develop surface and contour plots to understand the interaction effect of die temperature and feed moisture content on the quality attributes. A further objective is to understand the effect of die diameter and steam conditioning on pellet quality in a pilot-scale pellet mill.

2. Materials and methods

2.1. Materials

Nine bins of distiller's dried grains with solubles (DDGS) received from a local ethanol factory were used for pelleting studies. The DDGS was collected from the factory at one time and stored for further analysis. Moisture content (wet basis) and bulk density of DDGS received were in the range of 8.56–10.72% and 357–398 kg m^{-3}. AACC (1995) method was used for moisture measurement and bulk density was measured based on procedure given by Canadian Grain Commission (2009). Bins #1–4 had moisture contents less than 9% and bins #6–9 had moistures of 10% or higher. Further, bins #1–4 were clustered into one group and bins #6–9 as another group. Particle size analysis (procedure described in Section 2.6.3) of the DDGS has indicated that the geometric mean diameter of DDGS was 0.54 ± 0.34 mm.

2.2. Proximate analysis

The proximate analysis of the initial DDGS was done at the Department of Animal and Poultry Science, University of Saskatchewan, Saskatoon, SK. Samples for the proximate analysis were made into three groups. Group-1 includes samples from bins # 1–3, Group-2, bins # 4–6 and Group-3, bins # 7–9. Dry matter (DM) was determined according to methods of the Association of Official Analytical Chemists (AOAC, 1999; ID 930.5). The crude protein (CP) content was determined using the AOAC standard method (AOAC, 2001), where the nitrogen content was multiplied by a factor 6.25. Neutral detergent fibre (NDF) was determined using AOAC standard method 992.16 (AOAC, 1999b). Acid detergent fibre (ADF) was determined using AOAC standard method 973.18 (AOAC, 1999a). Neutral (NDICP) and acid (ADICP) detergent insoluble crude protein were determined by analyzing NDF and ADF residues for Kjeldahl nitrogen (Licitra, Mernandez, & Van Soest, 1996). Neutral detergent residual crude protein (NDRCP) and acid detergent residual crude protein (ADRCP) were estimated by the procedures given by Goering et al. (1972). All the calculations were made on DM basis.

The results from the proximate analysis (Table 1) were analyzed statistically using one-way analysis of variance and the means were compared using Scheffe's method which is a single-step multiple comparison procedure. This method basically works on adjusting significance levels in a linear regression analysis to account for multiple comparisons. It is particularly useful in analysis of variance, and in constructing simultaneous confidence bands for regressions involving basis functions. In the present study, the Scheffe procedure was used for multiple comparisons as it allows pair wise comparison and combinations of means. In general, if the results are significant by Scheffe, it is likely that they will be significant by other procedures also (Aparna, McCann, & Edwards, 1998; Khuri & Cornell, 1987).

2.3. Moisture adjustments

The moisture to be added or removed from the DDGS for carrying out the pelleting trials was estimated by assuming that the weight of the DM remains constant.

2.3.1. Preconditioning of distiller's dried grains with solubles

The DDGS from bins which had to be pelleted with no steam was adjusted to 13.5% moisture. DDGS samples were initially put in a plastic pail fitted into a cement mixer. The calculated amount of additional water was sprayed onto the samples. These samples were stored overnight at ambient room temperature to allow for equilibration of the moisture.

2.4. Pelleting studies using the single pellet machine

A single pelleter was used to study the compression characteristics of DDGS. The pelleter, which included a heating element, had a diameter of 6.35 mm and length of 135.34 mm and was insulated. Two T-type thermocouples were used, with one near the die wall and other near the heater. The thermocouple near the die wall was connected to a temperature controller. The pelleter was fitted on a stainless steel base with a hole matching the outer base diameter of the pelleter. Loading was made possible by a 6.35 mm plunger attached to an Instron (Model 1011 testing machine, Instron Corp., Canton, MA) equipped with a 5000 N load cell (Tabil, 1996). The preset load used for the test was 4500 N at a cross-head speed of 50 mm min^{-1}.

The amounts of moisture to be added or removed during single pelleting studies were calculated using mass balance. The moisture-adjusted DDGS was sealed in air tight containers and stored in a cold store for equilibration. A sample of about 0.5–1.0 g was fed into the heated die and compressed up to the specified preset load and held for 60 s to arrest the springback effect. The force–deformation data during compression and the force–time data during stress relaxation were logged in the computer. The pellet formed was removed by gentle tapping using a plunger. After each trial in the single pellet machine, pellet properties (mass, length, diameter, pellet moisture content, durability and pellet density) were measured. Each test was repeated five times. The die was heated in the temperature range of 50–100 °C in order to simulate the heating in commercial pelleting mills. Moisture content was varied between 5.1, 9.0 and 11.8%.

2.5. Pelleting studies using a pilot-scale pellet mill

2.5.1. Pilot-scale pellet mill

A pilot-scale pellet mill (CPM-Laboratory Model CL-5, California Pellet Mill Co., Crawfordsville, IN) was used to produce pellets using DDGS. The schematic diagram and pellet mill details are discussed by Adapa, Tabil, Schoenau, and Sokhansanj (2004) in their studies on pelleting of sun-cured and dehydrated alfalfa grinds. The pellet mill is provided with a receiving hopper and vibratory feeder to hold the DDGS and regulate the flow of the DDGS into the pellet mill. The pellet mill is connected to a steam supply line where the line pressure was reduced from 965 kPa to between 69 and 345 kPa using a pressure reduction valve. A 0.8 mm diameter nozzle was used to introduce the superheated steam into the conditioning chamber to moisten and heat the DDGS. The blades of the paddles are arranged in a screw like manner with respect to whole length of the shaft and were oriented at an angle of 7°. At this orientation the residence time of the DDGS in the chamber is about 17–20 s (Adapa et al., 2004; Tabil, 1996).

The pellet mill was instrumented and interfaced with a personal computer to record temperature and pellet output rate. Temperature sensors were placed at different positions

Table 1 – Proximate composition of wheat-based distiller's dried grain with solubles.

DDGS sample	DM (%)	CP (%)	NDF (%)	NDRCP (%)	NDICP (%)	ADF (%)	ADRCP (%)	ADICP (%)
Bin 1–3 (Group-1)	92.60[c]	40.33[a]	51.04[a]	24.48[c]	60.69[b]	22.93[b]	9.67[b]	23.98[b]
Bin 4–6 (Group-2)	92.38[b]	37.64[a]	51.79[a]	21.97[b]	58.37[b]	22.33[b]	8.27[b]	21.99[b]
Bin 7–9 (Group-3)	91.27[a]	39.93[a]	51.87[a]	19.05[a]	47.71[a]	15.78[a]	3.61[a]	9.07[a]

Note: (1) Means on the same column with the same letters are not significantly different at 5% level. (2) The measured values are average of three measurements. (3) The minimum and maximum SD values were 0.2–0.23.
Note: DM: Dry matter; CP: Crude protein content; ADF: Acid detergent fibre, NDF: Neutral detergent fibre, NDRCP: Neutral detergent residual crude protein; NDICP: Neutral detergent insoluble crude protein; ADRCP: Acid detergent residual crude protein, ADICP: Acid detergent insoluble crude protein.

including in the steam line, in the hopper discharge to measure the pre-conditioned DDGS temperature, in the conditioner at regular intervals, at the receiving hopper of the screw feeder to measure the conditioned DDGS temperature, and at the output container to measure pellet temperature (Adapa et al., 2004; Tabil, 1996).

2.5.2. *Pelleting*

The 1.5 kW (2.0 hp) CPM CL-5 pilot-scale pellet mill was used for continuous pelleting studies. Two die diameters, 6.4 and 7.9 mm with length-to-diameter (*L/D*) ratios of 7.3 and 4.1, were used. Pelleting was conducted with 6.4 and 7.9 mm die without steam addition but DDGS moisture was adjusted to 13–14% (w.b.). Also, pelleting was done with 6.4 and 7.9 mm die with steam addition using DDGS 'as received'. DDGS samples from bins 1, 6, 3 and 8 were pelleted without steam conditioning and those from bins 2, 4, 7 and 9 with steam conditioning.

During pelleting using 6.4 mm die with steam addition, the heating pads in the conditioner of the die were also heated to provide additional heat to the samples. During pelleting with 7.9 mm die, the heating pads of the conditioner were heated both for steam and without steam addition. Pellets were not formed in 7.9 mm die when the heating pads were off when no steam was added. When using the 7.9 mm die, additional moisture was added to the DDGS to reduce dust generation and to increase pellet formation. The pellet mill was run at a speed of 316 rpm. The flow of the DDGS was controlled by a vibratory feeder located at the hopper outlet. The pellets were collected from the outlet of the pellet mill. The throughput of the pellets from the pellet mill was determined by collecting amount of the pellets for a known period of time. The pellets were spread on plastic bags and then cooled to room temperature (22–24 °C). The cooled pellets were placed in plastic bags and put in cold storage at 5 °C and were further used to measure bulk density and durability. Moisture content of the pellets was determined immediately after pelleting and after cooling. Durability and bulk density of the pellets were also measured.

2.6. *Measurement of physical properties of granular and pelleted distiller's dried grains with solubles*

2.6.1. *Moisture content of granular distiller's dried grains with solubles*

The moisture content of DDGS was measured based on AACC (1995) method, where 2–3 g of samples was dried at 130 °C for 1 h and the moisture is expressed in % wet basis (w.b.). The reported values are average of three measurements.

2.6.2. *Moisture content of pellets*

The moisture content of the DDGS pellets was determined in accordance with ASAE S358.2 method (ASAE, 2003). About 25 g of the pellets were placed in an oven at 103 °C for 24 h. Moisture measurements were done in triplicates.

2.6.3. *Particle size analysis of distiller's dried grains with solubles*

Particle size analysis of the DDGS was carried out by sieving. Sieving was done in two stages. In the first stage, sieve numbers 10, 12, 20, 30, 40, 50 and 60 were used and in the second stage, sieves with finer openings (US No. 70, 80, 100, 140, 200, and 275) were used. The sieving test lasted for 10 min using a Ro-Tap The sieving shaker.

For the first stage, 100 g of DDGS was used, in three replicates. At the end of first stage sieving, DDGS retained in the sieves and in the pan was weighed. Those retained in the pan were further used for the second stage sieving. The geometric mean diameter was calculated using ANSI/ASAE S319.4 method (ASABE, 2008).

2.6.4. *Pellet density*

The density of each individual pellet (pellet density) was calculated by measuring the length and diameter using electronic caliper and the mass using an electronic balance with 0.01 g precision. To have uniform length, the edges of the pellets were smoothed. Pellet density was calculated by dividing mass of individual pellets by their volume calculated from length and diameter (Shankar et al., 2007, 2008). Pellet density values reported are an average of five measurements.

2.6.5. *Bulk density of granular and pelleted distiller's dried grains with solubles*

Bulk density was measured based on the procedure given by the Canadian Grain Commission (2009). A container (0.5 l) was used to determine the volume of the sample. The pellets were first sieved using a 5.56 mm round hole. The container was first weighed and the samples were dropped from a height of 50.8 mm. The surface of the material was levelled flush with the container and the total mass of the material and the container was measured. Bulk density was determined by expressing the ratio of the mass of the material to the volume. The reported values are an average of five measurements.

2.6.6. *Durability measurement of a single pellet (drop test)*

Durability of pellets produced in single pelleting tests was carried out using drop test. Single pellets were dropped from a 1.85 m height on a metal plate. The mass retained is expressed as the percentage of the initial weight (Al-Widyan & Al-Jalil, 2001; Khankari, Shrivastava, & Morey, 1989; Sah, Singh, & Agrawal, 1980; Shrivastava, Shrivastava, & Khankari, 1989). Each drop test was replicated five times.

2.6.7. *Pellet durability measurement using a tumbler*

The durability of the DDGS pellets produced by the pilot-scale pellet mill was determined according to ASAE Standard Method S269.4 (ASAE, 2004). The pellets were cooled to room temperature (22 °C) and about 500 g was used for each test. Prior to testing, the pellets were sieved using round hole sieves with screen sizes of 5.6 and 6.7 mm corresponding to the pellets made using die diameters 6.4 and 7.9 mm, respectively. The pellets were tumbled inside a pellet durability tester for 10 min. The pellets were sieved after tumbling and the durability index was calculated as ratio of mass of pellets remaining on a sieve after tumbling to the initial mass before tumbling. The reported values are an average of three measurements.

2.7. *Statistical modelling and analysis*

2.7.1. *Regression models*

Second-order regression equations were developed for die temperature and feed moisture content in terms of response variables pellet moisture content, pellet density and durability. Eq. (1) indicates the form of the second-order polynomial used in the present study for modelling of pellet density and pellet moisture content (Khuri & Cornell, 1987) and a second-order polynomial without the quadratic terms was used to model the durability data.

$$y = b_0 + \sum_{i=1}^{n} b_i x_i + \sum_{i=1}^{n} b_{ii} x_i^2 + \sum_{i=1}^{n} \sum_{j=1}^{n} b_{ij} x_i x_j + \varepsilon \quad (1)$$

where y is the dependent variable (observed), x_i and x_j are the coded independent variables, b_0, b_i, b_j and b_{ij} are coefficients, n is the number of independent variables and ε is a random error. The second-order polynomial in general is not a true representation of the process, but still yields good results for process and product development in chemical and biological engineering problems (Khuri & Cornell, 1987).

2.7.2. *Analysis of variance and response surface plots*

Analysis of variance (ANOVA) was carried out to assess the effect of the independent variables tested (linear, quadratic and interactive) on the dependent variables pellet moisture content, pellet density and durability. Sheffe's test was used to study the significant effects of treatments used in the pelleting process.

Response surface plots were drawn for the two independent variables die temperature (T) and feed moisture content (M_w) for pellet moisture content, pellet density and pellet durability to understand the trends of the process variables which can result in optimization of the quality attributes.

3. Results and discussion

3.1. *Proximate analysis of distiller's dried grains with solubles*

The proximate analysis of the DDGS (Table 1) indicated that the average CP and DM were in the range of 37.37–40.33% and 91.27–92.60%. Statistical analysis of the DDGS composition data using Scheffe's method indicated that there was no significant difference in the CP content measured in the three groups but the DM content was found to be significantly different at $P < 0.05$. No significant difference was observed between Groups 1 and 2 for the other DDGS composition data like ADF, NDF, NDRCP, NDICP, ADRCP, and ADICP except for Group-3 which was significantly different at $P < 0.05$.

3.2. *Single pellet test results*

Table 2 show the results of the single pellet tests carried out at three levels of die temperatures and feed moisture contents. Higher pellet densities, lower pellet moisture content and higher durability are achievable at low feed moisture content and high die temperature. During the compression of the DDGS at high temperature (Table 2), there was a significant loss of moisture from the sample due to heating and this is reflected in the measured pellet moisture content values. Table 2 data were further used for developing the regression equations, ANOVA analysis and response surface plots.

3.2.1. *Equations and analysis of variance*

The regression equations expressing pellet moisture content, pellet density and durability as functions of die temperature and feed moisture content are given below:

$$\mathrm{PMC} = -4.73 + 0.077T + 1.20M_w - 0.000451T^2 - 0.033M_w^2 - 0.0027T \times M_w \quad (2)$$

$$\mathrm{PD} = -1216.1 + 4.03T - 67.38M_w - 0.031T^2 + 0.49M_w^2 + 0.185T \times M_w \quad (3)$$

$$D = 111.384 - 0.11T - 1.869M_w + 0.0678T \times M_w \quad (4)$$

where PMC is pellet moisture content (%), PD is pellet density (kg m^{-3}), D is pellet durability (%), T is process temperature and M_w is the feed moisture content.

Based on the coefficient of multiple determination (R^2) value and the plot of predicted and observed values, the regression equations for pellet moisture content, pellet density and durability were adequately described by the process variables, die temperature and moisture content. The overall significance of all the models developed was found to

Table 2 – Experimental results of pellet moisture content, density and durability from single pellet tests.

Independent variables		Dependent variables		
Die temperature (°C)	DDGS initial moisture content (% w.b.)	Pellet moisture content (% w.b.)	Pellet density (kg m^{-3})	Pellet durability (%)
50	5.10	2.68	1056.1	99.9
75	5.10	2.70	1080.4	99.9
100	5.10	2.45	1071.3	99.7
50	9.00	4.99	860.00	98.6
75	9.00	4.60	899.60	98.1
100	9.00	4.42	903.10	98.4
50	11.80	5.83	719.10	91.4
75	11.80	6.06	782.50	98.3
100	11.80	4.67	797.30	97.2

Note: (1) Pellet moisture content was measured in triplicates; pellet density and durability are average of five measurements. (2) The measured SD values for pellet moisture content, pellet density and durability are in the range of 0.01–0.15, 15–25 kg m^{-3} and 0.1–0.9%.

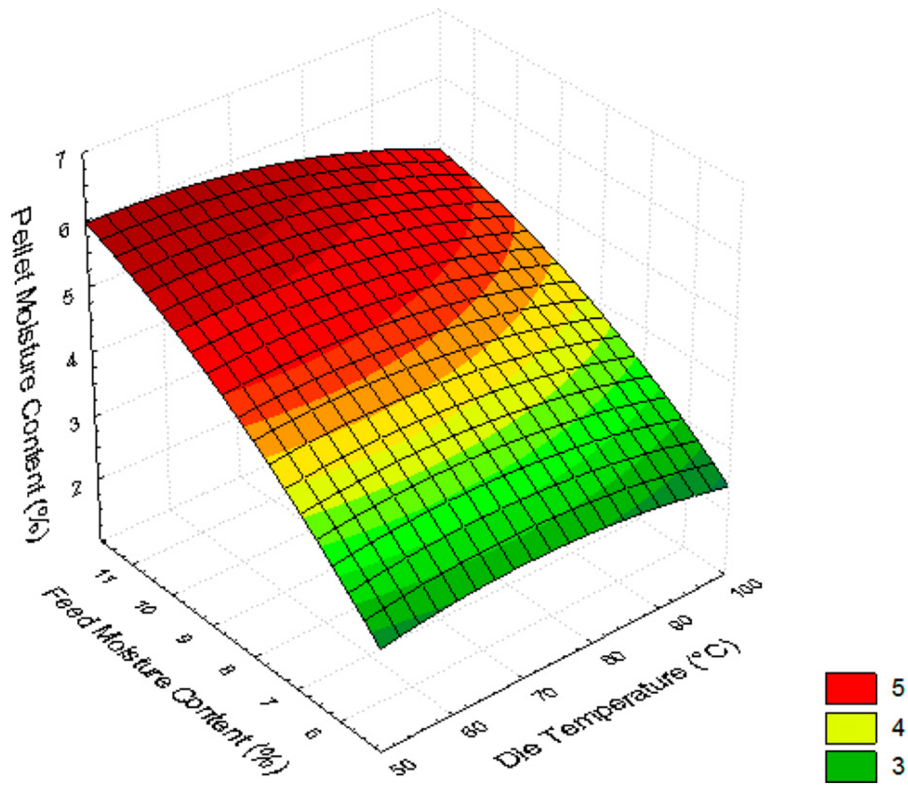

Fig. 1 – Effect of die temperature and feed moisture content on pellet moisture content (%).

be high ($P < 0.01$) for pellet moisture content and pellet density and $P < 0.05$ for durability. It was observed that the durability equation had lower R^2 value compared to pellet moisture content and pellet density. Many researchers have indicated that the durability values obtained using drop tests are not as consistent as the values measured using a tumbler. Also, the narrow range of measured durability values could have contributed to the lower R^2 value.

Based on ANOVA, the linear terms of T and M_w, the quadratic term of T and the interactive term $T \times M_w$ were

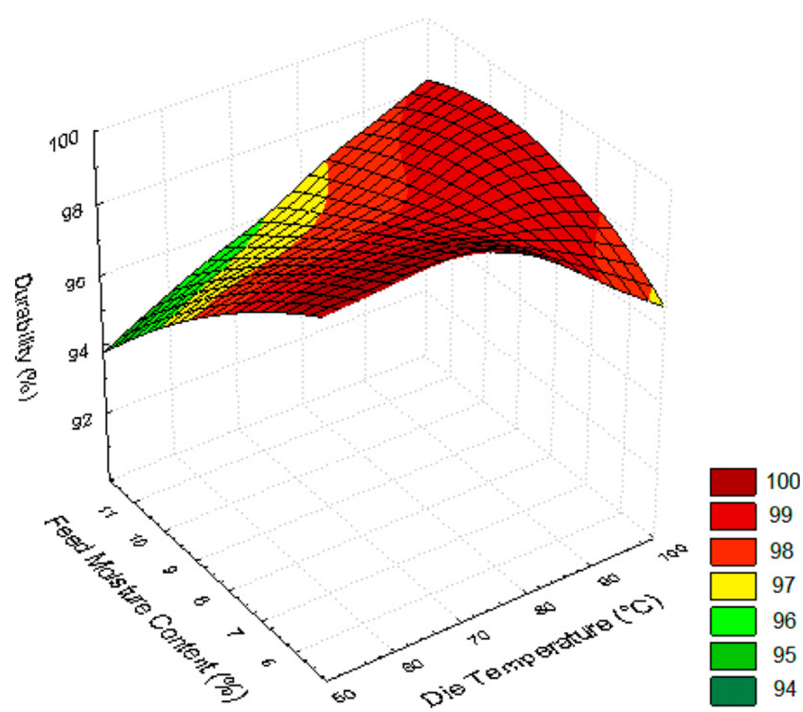

Fig. 2 – Effect of die temperature and feed moisture content on durability (%).

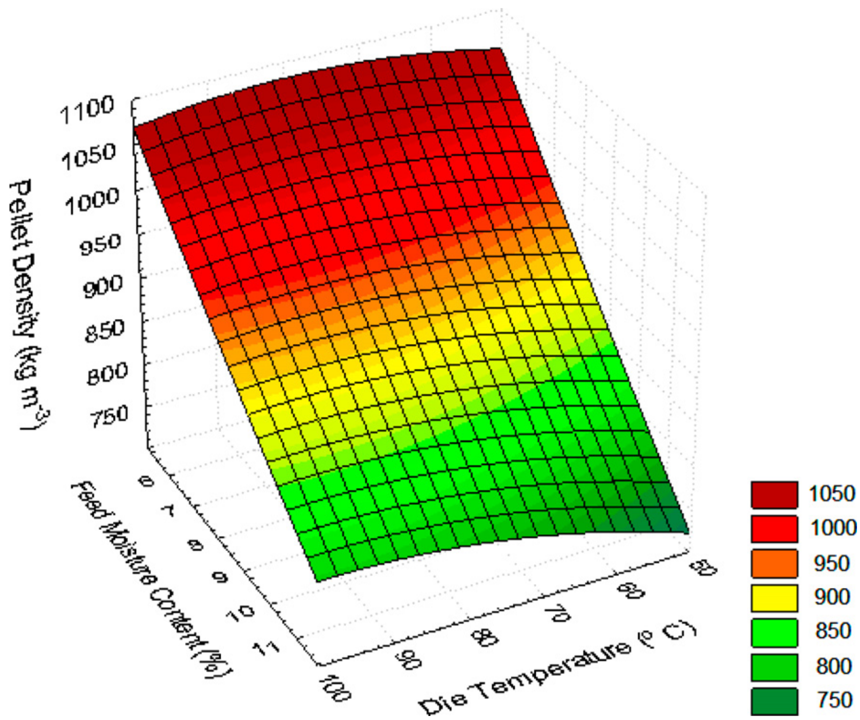

Fig. 3 – Effect of die temperature and feed moisture content on pellet density (kg m^{-3}).

found to be significant at $P < 0.01$ for pellet density. In the case of pellet moisture content, only the linear terms of T and M_w were found to be significant at $P < 0.1$; while in the case of durability, the linear term of M_w and interactive term $T \times M_w$ were found to be significant at $P < 0.1$.

3.2.2. *Response surface plots*

The surface plots were drawn for die temperature and feed moisture content for the response variables pellet moisture content, pellet density and pellet durability in order to understand the trends for optimization of process variables.

The surface plot (Fig. 1) for pellet moisture content indicated that an increase in feed moisture content significantly increased the pellet moisture content. The effect of die temperature was found to be marginal. A maximum pellet moisture content of about 5% could be reached at low die temperature of about 50 °C and high feed moisture content of about 11%. Lowering the feed moisture content to about 5% and increasing the die temperature to >80 °C resulted in the lowering of pellet moisture content to about 2.5%. Shankar et al. (2009) in their studies on the effect of process variables on extrudate moisture content reported that feed moisture content and barrel temperature had significant effects. They observed that high barrel temperature and low feed moisture content resulted in low extrudate moisture content. Falcone and Phillips (1998) in their studies on extrusion of snack products observed that extrudate moisture is inversely proportional to barrel temperature and directly to feed moisture content (Fig. 1).

Fig. 2 shows the response surface plot for the durability indicating that higher die temperatures and lower feed moisture content resulted in more durable pellets. Die temperature of 50–90 °C and feed moisture content of 5–7% resulted in maximum durability of about 100%. High temperatures of about 90–100 °C and high pressures of 4500 N may have resulted in partial gelatinization of little starch available in DDGS at low moisture contents of about 5–7% which in turn could have improved the durability values. Enterline (1982) observed that high pressure and temperature during processing of starch reduces the gelatinization moisture content. Mercier and Feillet (1975) in their studies on extrusion of starch indicated that high pressure and shear results in increased starch susceptibility to α-amylase degradation at low moisture contents. Many researchers have demonstrated that the extent of starch gelatinization is a strong function of barrel or die temperature and feed moisture content during extrusion of high starch food/feed materials (Bhattacharya &

Table 3 – Trends of process variables based on response surface plots from single pellet tests.

Quality attribute	Die temperature, T (°C)	Feed moisture content, M_w (%)	Objective
Pellet moisture content (%)	100	5.1	Minimize
Pellet moisture content (%)	50–80	11.8	Maximize
Durability (%)	<70	11.8	Minimize
Durability (%)	50–100	5.1–7	Maximize
Pellet density (kg m^{-3})	<70	11.8	Minimize
Pellet density (kg m^{-3})	>70	5.1	Maximize

Table 4 – Throughput, bulk density and durability of pellets produced using a pilot-scale pellet mill.

Bin #	Die diameter (mm)	Pellet throughput rate (kg h^{-1})	DDGS pellets bulk density (kg m^{-3})	Durability (%)	Percent increase in bulk density
No steam addition					
1	6.4 (NS)	45.52^{a*}	514.2^{de*}	68.0^{b*}	42.8
6	6.4 (NS)	50.34^{ab}	483.2^{c}	60.3^{a}	23.2
3	7.9 (NS)	64.56^{ab}	502.8^{d}	82.2^{d}	41.1
8	7.9 (NS)	90.88^{b}	436.8^{a}	74.3^{c}	9.7
With steam addition					
2	6.4 (S)	59.2^{ab}	528.9^{f}	92.7^{g}	45.7
7	6.4 (S)	68.77^{ab}	516.2^{e}	92.6^{g}	44.4
4	7.9 (S)	44.32^{a}	458.5^{b}	84.7^{e}	28.1
9	7.9 (S)	42.65^{a}	481.4^{c}	89.8^{f}	32.1

Note: (1) *Means with the same letter in the same column are not significantly different from each other at 5% level of significance. (2) S = with steam; NS = no steam.

Hanna, 1987; Cai & Diosady, 1993; Chiang & Johnson, 1977; Colonna, Doublies, Melcion, Demonredon, & Mercier, 1984; Diosady, Paton, Rosen, Rubin, & Athanassoulias, 1985; Guy & Horne, 1988; Lawton, Henderson, & Derlatke, 1972; Shankar and Bandyopadhyay, 2004).

The surface response plot for pellet density (Fig. 3) indicates that lower feed moisture content of about 5.1% and higher die temperature of about 100 °C resulted in maximum pellet density of about 1070 kg m^{-3}. It is also evident from the plot that feed moisture content has a more significant effect. Many researchers have observed that lower feed moistures and higher barrel temperatures during extrusion results in better texturization of the product which results in higher extrudate densities. Shankar and Bandyopadhyay (2005, 2006), and Shankar, Sokhansanj, Bandyopadhyay, and Bawa (2008a) in their studies on co-extrusion of fish and rice flour co-extrudates observed that high barrel temperature and low and medium feed moisture content result in better texture. Table 3 details the summary of the RSM plots, indicating the process conditions that can result in maximum and minimum values of pellet moisture content (%), pellet density (kg m^{-3}) and pellet durability (%).

3.3. *Pilot-scale pellet tests*

3.3.1. *Effect of steam conditioning on granular distiller's dried grains with solubles*

The moisture content of the DDGS after steam conditioning in the pellet mill increased from 8.5 to about 12% but no significant change in the bulk density was observed.

3.3.2. *Effect of steam conditioning on the pelleted distiller's dried grains with solubles quality*

The granular DDGS material was pelleted in the pilot-scale pellet mill with and without steam conditioning. Table 4 lists the throughput rates, moisture content and durability of the DDGS pellets with and without steam conditioning. It was observed that DDGS could be easily pelleted using 6.4 and 7.9 mm die without steam conditioning and the throughput rates were found to vary between 45.5–50.3 and 64.6–90.9 kg h^{-1}, respectively. However, the durability values measured for these pellets were in the lower range of 60–68%, indicating that the pellet may disintegrate easily either during storage or transportation due to poor binding of particles.

Pelleting of steam-conditioned DDGS using 6.4 mm die has given good results in terms of durability. The throughput rate observed was between 59.2 and 68.77 kg h^{-1}, much higher than the unconditioned samples. Durability values measured were also found to be the highest (Table 4). These pellets are less likely to disintegrate and will generate less dust during handling, storage and transportation. The steam-conditioned DDGS pelleted using the 7.9 mm die had lower throughput rate compared to unconditioned, but the durability values observed were higher. The statistical analysis of the results using Scheffe's multi-comparison test indicated that steam conditioning and die diameter have significant effect on pellet bulk density and durability at $P < 0.05$. Table 4 shows the percent increase of bulk density as a result of pelleting. Steam-conditioned DDGS when pelleted produced the highest bulk density using both die sizes. It was also observed that the pellets made with the 6.4 mm die produced had the highest bulk density with 45.7% increase from the initial bulk density value.

4. Conclusions

The present research was carried out to understand the effect of pelleting process variables on the quality attributes of wheat-based DDGS pellets. The following conclusions were drawn.

1. The linear regression models developed for the pellet quality attributes like pellet moisture content, pellet density and durability in terms of die temperature (T) and feed moisture content (M_w) adequately described the single pelleting process with R^2 value of 0.97, 0.99 and 0.70, respectively.
2. Based on analysis of variance, both the process variables T and M_w were found to be statistically significant at $P < 0.01$ for pellet density and $P < 0.1$ for pellet moisture content and durability.
3. The trends of the surface plots indicated that a medium temperature (about 50–80 °C) and low M_w (about 5.1%) have resulted in the maximization of pellet density and durability and minimization of pellet moisture content.

4. Both die diameter and steam addition were found to have significant effect at $P < 0.05$ on the bulk density and the durability of DDGS pellets.
5. DDGS pellets manufactured with a die diameter of 6.4 mm and with steam addition produced pellets with high bulk density and durability values compared to 7.9 mm die with or without steam addition.

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