

## Strength of Wheat Gluten Pellets Made at Different Temperatures, Moisture Contents and Compacting Stresses

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### ABSTRACT

The present article describes the effect of temperature, moisture content and compacting stresses over the density and strength of pellets using a laboratory pelleting equipment. The maximum tensile stress of wheat gluten pellets is increased by increasing the levels of moisture and temperature within the experimental range. However temperature, moisture content and compacting stress did not affect pellet density.

### INTRODUCTION

Wheat gluten (WG) is well-known for its functional benefits in various food applications. In recent years, it has been identified that WG's unique characteristics and functional properties can be utilized in the animal and fish feed industry. As a result, many new and novel applications and formulations within these industries have been developed.

WG is used as a protein source and as binding ingredient in pet foods and animal feed. Today, WG is a common protein source utilized as a supplement for fish feed<sup>1</sup>. WG is highly digestible and it has high energy content<sup>2</sup>. Also, WG has good palatability when used in diets with up to

40% inclusion into the fish meal in salmon or trout<sup>3</sup>. Furthermore, WG is used to feed other aquatic species such as marine shrimp<sup>4</sup> and juvenile lobsters<sup>5</sup>.

In pet foods, WG is used because of its excellent nutritional value and digestibility levels. The pet food industry ranks as the third user of WG which is equivalent to a 8% usage at worldwide level<sup>6</sup>.

Regarding the physical quality of animal and fish feed, WG increases the physical strength of pellets. It's been reported that increasing levels of WG in the diet up to 10%, noticeably increased pellet quality and water stability<sup>7</sup>.

The main drawback of WG is its relatively high price. WG is currently produced for human consumption as a high-value, non-meat protein source. However, as a lower feed grade quality, WG has a vast potential as a valuable non-animal protein source. Another characteristic of WG, but nutritionally speaking, is that has low contents of lysine and therefore it cannot be fed alone as the only feed source<sup>8</sup>.

Regarding the effects of WG in feed processing, WG cannot be used alone due to problems during extrusion process. WG produce instabilities in the die pressure and

extruder throughputs and thus steady state is difficult to achieve<sup>9</sup>.

WG has been described as viscoelastic in the presence of water<sup>10</sup>. Evidence obtained from spectroscopic analysis has led to the suggestion that hydrogen bonding between repeated regions of high molecular weight subunits is responsible for the elasticity of gluten<sup>11</sup>.

## MATERIALS AND METHODS

### Experimental design

The experimental design was made considering a full factorial design. The different parameters are described as follows: four different temperatures (60, 70, 80 and 90 °C), six different compacting pressures (7, 9, 11, 13, 15 and 17 MPa), and five different moisture contents (11, 13, 15, 17 and 19 % w/w).

The temperature range was chosen to cover the most common pelleting temperatures of animal feed. The pressures chosen were selected to cover the most common pellet densities found in commercial animal feed from pelleting process. The moisture contents were also chosen to cover the most common moisture contents used in industrial feed production. In most cases of pelleting of animal feed, moisture contents over 18% begin to produce pelletability problems on ring type of die on pellet presses.

### Preparation of the raw materials

The preparation of WG was carried out at Centre for Feed Technology – FôrTek, an experimental feed plant associated to the Norwegian University of Life Sciences (UMB), Ås. This research was done using Cargill Vital wheat gluten powder (Table 1) (Cargill, Barby, Germany) which was provided in a 25 kg bag.

Table 1. Composition of the WG powder

Composition of WG*	(g kg <sup>-1</sup> )
Moisture content	78
Dry matter	922
Crude protein	776
Crude fat	50
Ash	12
Total carbohydrate	70
Starch	9

\* Data provided by Cargill, Barby, Germany.

The experimental design demanded a precise addition of small quantities of water. For this purpose, a six liter twin shaft paddle mixer / vacuum coater, model F-6RVC - 2001, Forberg – Landteknik, Norway, was used. The water was added by a pressurized tank (Teknisk Vannsevice AS, type Opür F-50; 1"20", model 2006) using approximately 5.5 bars of air pressure. The spray nozzle is commercially referred to as Uni-Jet 650025, which, according to the producer, delivers 130 g of water per minute at 5.5 bars of air pressure. The WG powder temperature before mixing was 8°C and the water temperature was 16°C. The moisture contents of WG powder used for the experiments were 11, 13, 15, 17 and 19 % w/w.

Immediately after water addition, the WG powder was collected and packed into sealed plastic bags to prevent moisture loss. The samples were stored in a cooling room at 4 °C for about three weeks until the pelleting process started.

### Pelleting method

All pellets used in this research were produced in a new laboratory die pelleting rig which is an upgrade of the laboratory pelleting rig used by Salas-Bringas et al.<sup>12-15</sup>. The rig was assembled in an Instron 100 kN texture analyzer. The new die pelleting rig consists of a cylindrical barrel made of construction steel having a compressing channel along the center, the compressing

channel is made from a stainless steel 321 pipe tightly assembled into the center of the barrel. The compressing channel has a diameter of 8 mm. A specially made tungsten carbide rod was used to press the samples against another rod located at the bottom of the compression channel. Using this configuration, the system can produce compacting stresses up to 400 MPa. To release the pellets from the compressing channel, the bottom rod was removed from the barrel. The same texture analyzer was used to provide the force needed to remove the pellet by pressing the upper rod.

The cylindrical barrel is heated by a jacket heater of 450 W which is controlled by a PID connected to a thermocouple type J attached to the barrel surface.

#### Production of pellets

At the beginning of the experiment, relatively long pellets were produced. It was found evidence of density gradients within the long pellets and this is discussed in the section presenting the results. The density gradient in long pellets stressed the need of making shorter pellets to avoid a gradient in the compaction stresses.

Approximately 0.15 g of WG was used to produce each short pellet. The sample was poured into the compressing channel and a pre load of 200 N (~ 4 MPa) was applied during 4 minutes of heating time. Following this time a slow compaction was applied at a speed of 0.25 mm minute<sup>-1</sup>. Immediately after the compaction process, the pressure was released and the pellet was removed from the pelleting rig. The pellets were stored in sealed plastic bags for the later strength tests under diametral compression.

#### Measurement of pellet strength and density

Measurements of strength for each pellet were obtained by measuring the first peak force ( $F$ ) in Newtons during a diametral compression. The tests were done

using a probe with a flat surface of 60 mm diameter which was connected to a Lloyd LR 5K texture analyser, the same testing arrangement has been used in previous research<sup>13-16</sup>. The compression speed was set to 0.25 mm minute<sup>-1</sup>.

The maximum tensile stress ( $\sigma$ ) for cylindrical specimens is estimated using Eq. 1<sup>16-19</sup>, which is commonly referred to as the “Brazilian” or “indirect” tensile test, as the tensile fracture is produced in a disc-shaped material by compressive loading across the diameter<sup>19</sup>.

$$\sigma = \frac{F}{\pi r L} \quad (1)$$

Where  $r$  and  $L$  are the radius (m) and length (m) of the pellets, respectively.

The density of the pellets was calculated from the volume of a cylinder since the pellets were shaped into a cylinder. The length, diameter and weight of the pellets were measured with a digital calliper and a scale respectively.

#### Effects of temperature, moisture and compacting pressure on density of pelleted WG powder

An ANOVA analysis using a general linear model of the main factors (temperature, moisture and compacting pressure) in Minitab software (Minitab Inc, USA) is used to see the effect on pellet density.

#### Effects of temperature, moisture and compacting pressure on strength of pelleted wheat gluten powder

Similarly as described for strength analysis, an ANOVA analysis using a general lineal model of the main factors (temperature, moisture and compacting pressure) in Minitab software (Minitab Inc, USA) is used to see the effects on pellet strength.

## RESULTS AND DISCUSSIONS

### Evidence of die friction and rheological changes

From Fig. 1, it is possible to visualize the effects of die friction during the compaction process. Previously, it has been mentioned<sup>13, 20</sup> that a source of error in this type of compression tests is that the friction between the material and the side walls of the die reduces the compaction stress from top to bottom (i.e. from the compressing rod to the opposite side of the channel). From Fig.1 it can be seen a color gradient in most of the pellets.

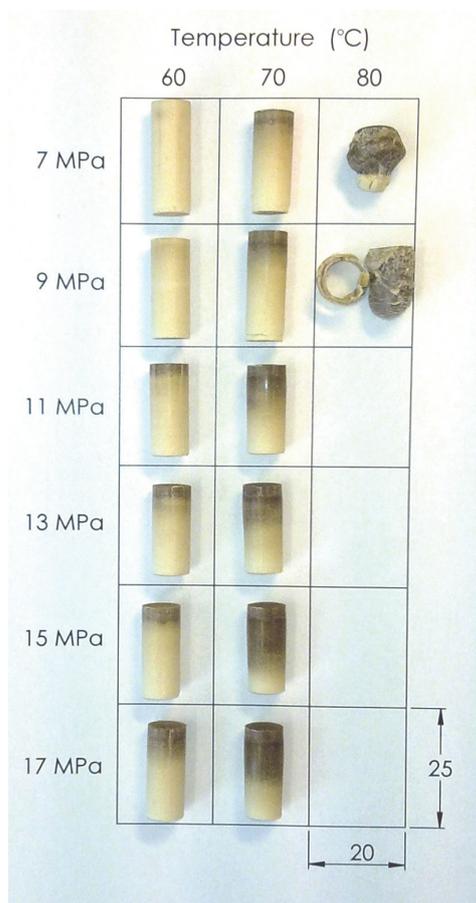


Figure 1. Evidence of pressure gradient at the die during compaction and phase transition after compaction of wheat gluten powder at different pressures and temperatures. All samples have 11 % moisture (w/w). Dimensions are in millimetres.

The whiter color of the pellets has a similar color to the uncompressed WG powder. The brownish color is more dominant at higher temperatures and moisture contents (Fig. 1 and Fig. 2). When pressed by the fingers and still warm, the brownish compacts showed a plasticized consistency, similar to the consistency of modelling clay. This softening change evidences the effects of a phase transition which can be observed from Fig. 3.

As mentioned in the previous section, the results from Fig. 1 stressed the need of making shorter pellets to avoid as much as possible the pressure gradient at the compacting rig. Consequently, all pellets made for the subsequent analysis were much shorter.

Fig. 2 is a visual example of the effects of moisture content in WG compacted at different pressures. Here were produced shorter pellets; the average length of the pellets shown in Fig. 2 was  $2.7 \pm 0.0005$  mm ( $\pm$  standard deviation). Similarly as found in Fig. 1, the pellets from Fig. 2 show a change in color at different moisture contents at 11 % w/w moisture, the pellet show a color change when compacting pressure was increased. These visual results also can be used as an evidence of a phase change. A temperature gradient in these pellets is neglected since the die was accurately warmed by a jacket heater connected to a PID controller, also the residence time of the powder inside the hot die before starting the compression was 4 minutes and the time that passes until the compacting pressure was released (i.e. dwell time) was about 7 minutes. The compaction speed was set to  $0.25 \text{ mm minute}^{-1}$  to minimize the heat generation due to die friction. Also, since the pellet have an average length of only 2.7 mm, temperature differences within a pellet can be neglected.

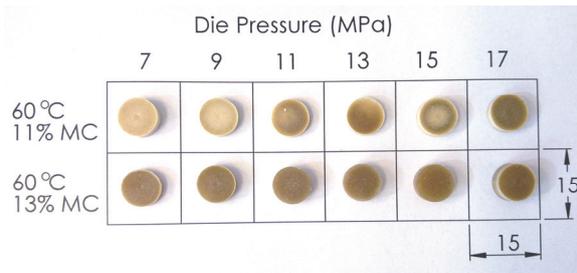


Figure 2. Evidence of rheological changes after compaction of wheat gluten powder at different die pressures and moisture contents (MC, % w/w). Dimensions are in millimetres.

Fig. 3 shows the glass transition temperatures ( $T_g$  (°C)) of native WG at different moisture contents reported by Micard et al.<sup>21</sup> using differential scanning calorimetry and by Pouplin et al.<sup>22</sup> using dynamical thermal analysis.

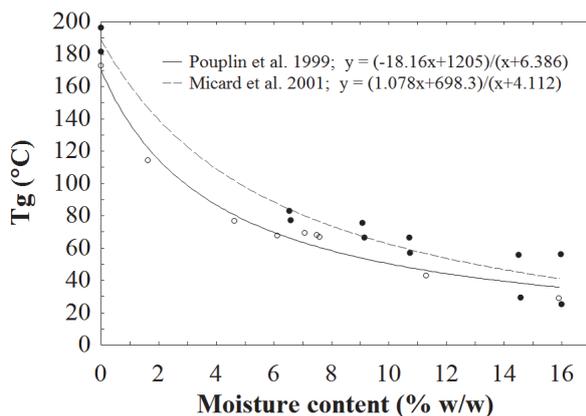


Figure 3. Glass transition temperatures ( $T_g$ ) of native wheat gluten at different moisture contents (% w/w). The data was re-plotted from Micard et al.<sup>21</sup> and from Pouplin et al.<sup>22</sup> New curve fits were made for comparisons.

According to the data from the curve fit made to Micard et al.<sup>21</sup>, at 11 % moisture (w/w),  $T_g$  of native WG is 47 °C and according to Pouplin et al.<sup>22</sup>, 57.5 °C.

According to Fig. 1 and Fig. 2, WG with 11% moisture (w/w) and 60 °C, presented a whiter color at low compacting stresses, however at higher compacting stresses the color of WG changes clearly to a brownish tone showing a possible phase change which can be ratified by Fig. 3.

The possible rheological and compositional differences between the WG used in this study with the WG used by Micard et al.<sup>21</sup> (vital wheat gluten, Amylum, Aalst, Belgium) and Pouplin et al.<sup>22</sup> (vital wheat gluten, Amylum Aquitaine, Bordeaux, France) have to be taken into account when comparing the data.

#### Effects of temperature, moisture and compacting pressure on density of pelleted WG powder

The results from the ANOVA analysis, showed that pellet density was not affected ( $p > 0.05$ ) by any of the main factors, temperature, compacting pressure and moisture content. This result can be explained by an elastic recovery of the pelleted WG. It is well-known that WG present a strong viscoelastic behavior in presence of water<sup>11</sup>. Previously<sup>23</sup>, have been stated that when die compaction finish, during pressure release, a relaxed elastic recovery should be expected from feed materials. However, an important part of it should be resisted by the presence of friction between the particles. Friction is present due to the existence of residual stresses within the compact that have a component that is normal to the die surface.

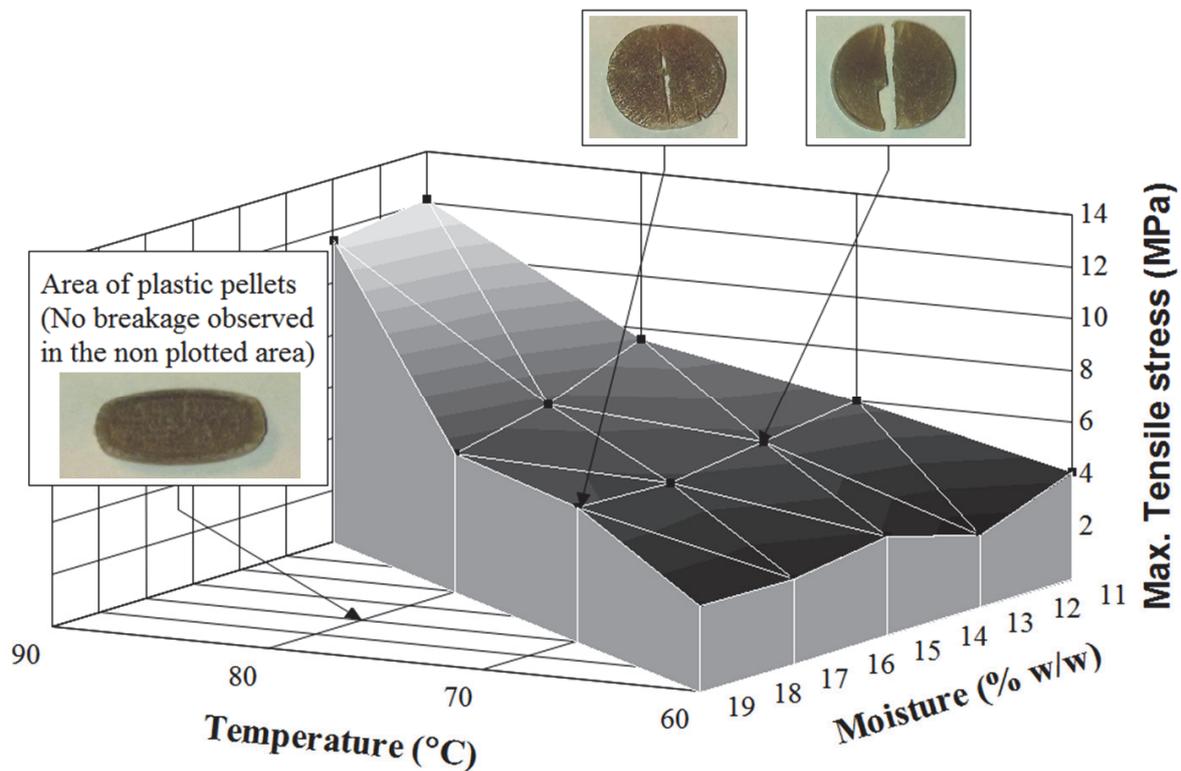


Figure 4. Maximum tensile stress (MPa) of wheat gluten pellets made at different temperatures (°C) and moisture contents (% w/w). The area where no breakage was observed represents the region of plastic pellets. Black dots indicate the average Max. Tensile stress (Mpa) (n = 6). The typical failure modes found in the pellets under diametral compression test can be seen inside the boxes.

Effects of temperature, moisture and compacting pressure on strength of pelleted wheat gluten powder

The results from ANOVA showed a significant ( $p < 0.001$ ) effect of temperature on the strength of WG pellets. Higher production temperature increased the strength of WG pellets ( $p < 0.001$ ). Also moisture content affected significantly ( $p \leq 0.001$ ) the strength of WG pellets. Higher moisture content decreased the strength of pelleted WG within the experimental range (11-19 % (w/w)). These results can be seen in Fig. 4. However, compacting pressure did not produce a significant effect ( $p > 0.005$ ) on pellet strength, reason why Fig. 4 plot the averages of the maximum tensile stresses of pellets made at different compacting pressures (black dots).

The equipment proves its capability to perform comparative results of pellets made

at different temperature, moisture and pressure conditions.

These types of analysis are not possible to perform using commercial pellet presses because pressure and temperature are the result of the feed material properties, flow rate, die profile and die friction. The results obtained from this research can be utilized in a pelleting process to see if WG is been pelleted within its maximum, minimum or medium range of strength.

**CONCLUSIONS**

The conclusions from this research shows that temperature, compacting pressure and moisture content do not produce significant ( $p < 0.05$ ) changes in the density of the pellets. However, temperature and moisture content affect significantly the strength of the pellets. Higher levels of moisture and temperature increased the tensile strength in the pellets.

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