Corn flour as affected by whole grain wheat flour and extrusion conditions: a study of physical and thermal properties and X-ray diffraction

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List of Abbreviations

FM Feed moisture
WGWF Whole grain wheat flour
SME Specific mechanical energy
WAI Water absorption index
WSI Water solubility index
RTE Ready-to-eat
CF Corn flour
Tp Gelatinization peak temperature
Keywords: amylose-lipid complex, crystallinity, extrusion, gelatinization, starch.

Abstract

Starch gelatinization is one of the most important transformations during the extrusion process. The objective of this study was to investigate the effect of extrusion variables (feed moisture-FM and temperature) and the incorporation of whole grain wheat flour (WGWF) on specific mechanical energy (SME) of the system and some properties related to starch changes (water absorption index, water solubility index-WSI, thermal properties and X-ray diffraction properties) using an experimental design. Non-extruded flour blends were also evaluated. Increasing WGWF and FM decreased both SME and WSI, reflecting decreased extrusion process severity. In extrudates, part of native crystalline structures rearranged from A-type to V-type after extrusion and formation of amylose-lipid complex took place. The differential scanning calorimetry (DSC) curves showed two gelatinization peaks for non-extruded flour blends (56.04-78.25 °C) and a third peak at higher temperatures (83.49-100.15 °C) attributed to amylose-lipid complex. The extrusion process promoted the complete gelatinization of the starches. The effect of WGWF addition on the thermal properties of the flour blends was noticeable. Starch transformation, as well as its functional properties, studied in flour blends and extrudates were affected by temperature and FM. Thus, despite of technological challenge to include whole grain wheat flour in expanded extruded formulations, maybe amylose-lipid complex formed strengthens the properties of extrudates as functional food.
1. Introduction

Food extruders induce thermo-mechanical changes of raw materials [1]. Extrusion cooking is responsible for gelatinization and degradation of starch and also for changing the extent of molecular associations between components [2]. The effect of an extruder on the material processed can be described by the system variables in terms of the energy input into the material (thermal or mechanical), which is defined by processing variables, such as temperature, screw speed, feed composition and moisture of the starting materials [3]. It is well documented that water content in combination with temperature has a significant effect on starch melting transformation [4] and consequently on quality of extrudates, such as ready-to-eat (RTE) expanded extrudates.

The interest in partial or total substitution of refined flours by bran or whole grains as ingredients in processed foods as a mean to improve mainly the fiber content is increasing [5]. However, their incorporation in extruded products is still limited due to the difficulty to overcome the changes caused by fiber on technological quality (expansion and texture) [6]. The understanding of starch transformations (structure and phase transitions) in a complex system containing a fiber component from whole grains would help to control the process and to obtain a food product of quality [7].

During extrusion cooking, the native structure of amylose is partially destroyed, and new crystalline ones, corresponding to the amylose-lipid complex, are formed [8]. Also, the amylose-lipid complex can be naturally present in starch [9]. This complex can promote important nutritional impact in extrudates due to reduced susceptibility of starch to digestion, and contribute to a lower glycemic index as reported in some studies [10, 11, 12]. The complex formation between amylose and lipids has an important influence on structure, texture and other functional properties of puffed extrudates [13,
Only very limited research has been done to study the amylose-lipid complex formation in expanded extrudates incorporated with whole grain flours [15].

Ready-to-eat breakfast cereals consumption has gained popularity amongst cereal foods. Traditionally, breakfast cereals are manufactured from refined corn or wheat flours due to their low price and high availability. Technologically, refined flours are preferred due to their bland taste and ease of processing compared to whole grain flours [16]. As a result, breakfast cereals are mostly composed of digestible carbohydrates (starch), and are essentially an energy source but nutritionally poor foods. In the food industry, this represents the possibility to apply extruded flours or, more specifically, to produce extruded products from whole grains with improved nutritional quality.

Considering extrusion cooking as a well-established industrial technology useful in grain processing and the changes that the components (starch, lipids, proteins) of the food system undergo, the technological and nutritional quality of foodstuffs can be modulated by both whole grain flour in replacement of refined flours and extrusion conditions. Differential Scanning Calorimetry (DSC) and X-ray diffraction analysis reveal valuable information for food development, when the extrusion conditions and parameters were set [17]. The aim of this study was to investigate the effect of temperature, feed moisture and whole grain wheat flour on extrusion process severity and degree of starch transformation evaluated by specific mechanical energy (SME) of the system, water absorption index (WAI), water solubility index (WSI), thermal properties and X-ray diffraction patterns of RTE extruded cereals. Non-extruded flour blends were also evaluated to assess the impact of the extrusion process, especially on expected starch gelatinization.

2. Materials and Methods
2.1. Materials

The corn flour (CF) and whole grain wheat flour (WGWF) used in this study were provided by Milhão Alimentos™ (Inhumas, GO, Brazil), described as Fecomix 425, and Anaconda™ mill (São Paulo, SP, Brazil), respectively. The CF was stored in plastic barrels. The WGF was vacuum packed in plastic bags and stored at -21 °C until use. The flours composition and particle distribution are presented in Table 1.

2.2. Methods

2.2.1. Sample preparation and extrusion cooking

Extrudates were prepared using a co-rotating ZSK 30 twin-screw extruder (Werner & Pfleiderer Corporation, Ramsey, USA). Feed rate (13 kg/h), screw speed (325 rpm) and temperature of zones 1 and 2 (75 and 100 °C, respectively) were fixed. Whole grain wheat flour (%), feed moisture (%) and barrel temperature of zones 3 and 4 (°C) varied according to the experimental design (Table 2). Before extrusion, each sample set composed by corn flour and/or whole grain wheat flour was weighed to give a batch of 2.5 kg and mixed (flour blends) with distilled water, according to the experimental design (Table 2), using a planetary mixer (Hypo, HB 12). The blended flour was tempered overnight at room temperature (25°C) to ensure a uniform hydration level of the feeding material. The barrel diameter D was 30 mm, and barrel length L 872 mm (L/D= 29.07). A circular die was used at the end of the extruder, with a diameter of 3.0 mm, and a knife with average speed of 110 rpm. The screw configuration used is described by Oliveira, Schmiele and Steel (2017) [18]. Afterwards, products were dried and stored in metalized bags, with light and moisture protection until further analysis.

2.2.2. Specific mechanical energy (SME)
For each trial, the motor torque displayed in the monitor panel was recorded along extrusion. The specific mechanical energy (SME) was given by Equation 1, and expressed in Wh.kg\(^{-1}\) [19].

\[
SME = \frac{\text{screw speed (rpm) } \times \text{ motor power (kW) } \times \text{ torque (\%)} \times \text{ maximum screw speed (rpm) } \times \text{ mass flow rate (kg.h\(^{-1}\))}}{100}
\]

(1)

where screw speed = 325 rpm; motor power = 4000 kW and maximum screw speed = 500 rpm.

2.2.3. Water solubility index (WSI) and water absorption index (WAI)

Water solubility index (%) and WAI (g/g) were determined according to the method described by [20]. An amount of 2.5 g sample (ground extrudate) was suspended in 30 mL of distilled water at room temperature, for 30 minutes, with intermittent shaking, and then centrifuged at 2200 x \(g\) for 10 minutes in a 204 NR centrifuge (FANEM, Brazil). The supernatant was transferred into an evaporating Petri plate with known weight and kept in an oven with forced air circulation, TE 394/2 (TECNAL, Brazil), at 105 °C, for four hours. The WSI was the weight of the dry solids in the supernatant expressed as percentage of the sample weight. Water absorption index was the weight of remnant sediment per unit weight of original dry solids. The analysis was carried out in triplicate and the results expressed in dry basis. Analysis of the flour blends (non-extruded) was also carried out.

2.2.4. X-ray diffraction (XRD)

Extruded samples were finely triturated to a powder (60 mesh), by using a mortar and pestle. The analysis procedure followed that described by [21] using RINT 2000 (Rigaku, Japan) by irradiating the sample with Cu, line K, L=1.542 A. The diffractograms were collected under the conditions of 40 kV, 30 mA, with the scanning...
angle 2θ set from 3° to 35° at a scanning rate of 1 °/min. The relative crystallinity was quantitatively estimated based on the relationship between the peak and total areas following the method of [22], using the software Microcal Origin software (version 7.5, Microcal Software Inc., Northampton, MA, USA). To determine possible changes in crystallinity due to the extrusion process, analysis of the unprocessed flours and flour blends (non-extruded) were also carried out [23].

2.2.5. Differential scanning calorimetry (DSC)

Ground extruded samples (mortar and pestle apparatus) passed through a 60 mesh (0.250 mm) sieve were evaluated for the thermal properties using a Pyris 1 DSC differential scanning calorimeter (Perkin Elmer, Boston, USA), previously calibrated with indium. An amount of 3.0 ± 0.1 mg of each sample was weighed in hermetically sealed stainless steel pans (100 μL; 30 bar), followed by addition of 9 μL water. Heating of each sample was carried out from 25 to 140 °C at 10 °C/min rate, using an empty pan as reference. Onset (T₀), peak (Tₚ), and conclusion (Tₖ) transition temperatures, as well as the enthalpy changes (ΔH), were determined using the Pyris 1 software (Perkin Elmer, Fremont, USA). To determine possible changes induced by extrusion, unprocessed flours and flour blends (non-extruded samples) were also analyzed, to assess the source of starch for the endothermic peaks. Two runs were carried out for each sample.

2.2.6. Statistical analysis

The experimental data were evaluated using the Response Surface Methodology (RSM) to investigate the effect of the extrusion process (temperature and feed moisture) and the flour characteristics (whole grain wheat flour replacement) on specific mechanical energy, water absorption index and water solubility index (dependent
variables), starch crystallinity (from XRD analysis) and thermal properties (from DSC analysis). A total number of 18 runs were defined, including four central points. A second-order polynomial regression model was established to fit the experimental data ($P<0.10$) for each response variable, as shown in Equation 2.

$$
y_i = b_0 + \sum_{i=1}^{3} b_i x_i + \sum_{i=1}^{3} \sum_{j=1}^{3} b_{ij} x_i x_j, \quad (2)
$$

where, $y_i$ is the response variable; $b_0$, $b_i$, and $b_{ij}$ are the regression coefficients for constant, linear, quadratic, and interaction regression terms, respectively; $x_i$ and $x_j$ are the coded values of the independents variables.

The response surface plots were generated as a function of two variables, keeping the third variable constant at the central value, based on the complete or re-parameterized regression models. A second-order polynomial regression model was established to fit the experimental data ($P < 0.1$) for each response variable. Regression coefficients of determination ($R^2$) and lack-of-fit (not shown) were considered to accept the regression model. $R^2$ higher than 0.70 was accepted.

3. Results and Discussion

3.1. Specific mechanical energy (SME)

The SME ranged from 84 to 154.67 Wh.kg$^{-1}$, similar to the values found by [24] (60-170 Wh.kg$^{-1}$) in a study with whole wheat extrudates working at a higher range of barrel temperatures (120 or 180 °C) and similar feed moisture (18 or 22 %). The SME is a good quantitative descriptor in extrusion processes of the extent of macromolecular transformations and interactions that take place, i.e. starch conversion, and consequently, the rheological properties of the melt [25]. Higher SME usually results in a greater degree of starch gelatinization, greater extent of starch molecular size reduction and extrudate expansion [26]. The regression analysis showed negative linear
effects \( (P<0.10) \) of WGWF and feed moisture on mechanical energy (Table 3). WGWF and moisture may have provided a reduction in the viscosity of the food melt in the extruder by changing the distribution of shear, mixing, mechanical heat and convective heat in the extruder and affecting motor torque and SME [27]. The viscous behavior of the melt is highly dependent on starch content [28], which decreases as the WGWF content increases, mainly as a consequence of the presence of fiber. An increase in SME is desired for expanding products because it increases the degree of starch gelatinization and favors bubble formation, yielding better density and texture [29]. The sectional expansion index of the extrudates decreased when WGWF and moisture increased [30]. Therefore, lower WGWF and moisture content would be recommended attending to SME values.

Neither temperature (76-144 °C) nor interactions effects were significant on SME (Table 3), although the temperature effect on SME has been reported [31]. Presumably, the temperature range used in the present study was not high enough to change the melt rheology (viscosity).

3.2. Water absorption index (WAI) and water solubility index (WSI)

The regression analysis results indicated that all three variables had effects on WAI. A negative linear effect of WGWF and positive quadratic effects of feed moisture and temperature were evidenced (Figures 1A, 1C and 1D). Water absorption depends on the availability of hydrophilic groups which bind water molecules and on the gel forming capacity of macromolecules [32], but results induced by WGWF revealed that the starch gel-forming capacity is more determinant on WAI than the presence of hydrophilic compounds such as fibers. The quadratic effect induced by water and temperature can be related to the increase in starch gelatinization [33]. The extrusion process increased the WAI compared to the initial raw materials, going from 2.71 g/g...
raw WGWF and 2.96 g/g raw corn flour, to values that ranged from 5.00 to 8.25 g/g extruded sample.

The predictive response model for WSI showed similar significance to WAI, also with a negative linear effect of moisture. There was no significant interaction between the independent variables (Table 3). The measurement of WSI is often used to assess the degree of transformation (dextrinization) of ingredients during extrusion and is related to molecules of low molecular weight in the extruded sample. Increasing WGWF or the water content in the feed significantly decreased ($P<0.10$) the WSI, meaning that less molecules (mainly carbohydrates of low molecular weight) were released and dissolved in water. The effect of WGWF on lowering WSI can be attributed to the presence of long chain molecules (soluble and insoluble fibers). The effect of water content on the water solubility of starch can be explained by its effect on viscosity, reducing shear. Maximum WSI values were observed at the axial points (76.4 and 143.6 °C) for temperature (Figures 1B and 1F). Increasing temperature would increase the degree of starch gelatinization and breakage depending of process severity, which could increase the amount of low molecular weight and/or soluble starch molecules, resulting in an increase in WSI [32, 23]. The results for WSI coincide with SME with respect to the negative effects of WGWF and feed moisture and that correlation has already been reported by [23]. The highest WSI values occurred under the most severe conditions (the greatest SME), due to the greater degree of gelatinization, disintegration of starch granules and possible depolymerization, resulting in an increase in soluble material [34].

The WSI of the flour blends before extrusion ranged from 11.47 to 28.24 %, whereas after extrusion they ranged from 23.1 to 37.0 %, indicating a significant change
in starch properties, making the structures more soluble. Similar to this, [32] found an increase in WSI for extruded whole wheat flour compared to the raw material.

Therefore, effects on WAI and WSI must be explained in great extent due to the starch-water interactions, which in turn will be influenced by moisture level in the feed stream, presence of other constituents, machine characteristics, residence time in the extruder, and screw configuration [35].

3.3. X-ray diffraction

The loss of organized crystalline structure of the starch granules on extrusion cooking was confirmed by the X-ray diffractograms (Figure 2A-2F) compared to the non-extruded flours (Figure 2G). The unprocessed flours and flour blends showed A-type crystallinity, typically found in cereals, having peaks at 15°, 17°, 18° and 23° 2θ [36], as shown in Figure 2G. The X-ray patterns of extruded cereals generally exhibited peaks at 7° and 13°, indicating the existence of V-type crystallinity [37, 38]. The samples from the central point (50 % WGWF; 19 % feed moisture; 110 °C) also presented a peak at 24° 2θ. [16] reported that A-type structure was disrupted and V-hydrate was formed upon extrusion of wheat and almond flours blend at barrel temperature above 90 °C. The loss of crystallinity during extrusion is caused by mechanical disruption of the molecular bonds due to the intense shear fields within the extruder [39]. Therefore, under extrusion at low moisture content (expanded products), a mixture of small amounts of gelatinized and melted states of starch, as well as fragmentation exist simultaneously [3].

The values of crystallinity were in the range of 16.97-23.81 % for the extrudates against 25.20-34.00 % for unprocessed flours and flour blends (Table 2). There was no effect of any variable studied (WGWF, moisture and temperature) (Table 3) on extrudate crystallinity. After extrusion, the A-type crystallinity was rearranged and V-
type was formed independently of WGWF ratio and extrusion conditions. The crystallinity in extrudates indicates the formation of a complex between amylose and endogenous lipids (amylose-lipid complex).

3.4. Differential scanning calorimetry

3.4.1. Gelatinization of unprocessed flours and flour blends

The DSC scans for the raw flours and flour blends are shown in Figure 3 and gelatinization properties values are presented in Table 4. The effect of WGWF addition on the thermal properties of the flour blends was noticeable, as shown by the DSC curves. Peaks detected in the DSC scans showed one peak for CF, whereas two peaks appeared for WGWF, and flour blends showed a combination of those peaks depending on the flour proportions in the blends. The second peak observed in WGWF and formulations containing WGWF was observed from 85 °C to 98 °C, and it may be associated to an amylose-lipid complex present in native starch granules of the raw material [40]. Corn starch has more uniform granules [41, 42]. For the flour blends, the first and second peaks correspond to moisture-mediated disorganization and melting of starch crystallites, responsible for starch gelatinization (order-disorder transition) [42]. Generally, the enthalpy variation associated to the second gelatinization peak showed low values (Table 3), which can be attributed to corn starch crystallites. The third peak (100 °C) observed for the unprocessed flour (except CF 100) and flour blends can be attributed to the dissociation of the amylose-lipid complex during the process [43]. There was a significant ($P<0.10$) difference between the enthalpy variation of peak 1 and peak 2 for most of the samples (Table 4). Corn flour had the highest $\Delta H$ for the gelatinization transition; the WGWF addition led to lower gelatinization enthalpy because of dilution of starch and less starch available for gelatinization, in the presence of fiber, which agrees with previous results [44]. The gelatinization peaks for 79.76 %
WGWF and 20.24 % WGWF were similar to the predominant flour in the blend, 100%
WGWF and 100% CF, respectively (Figure 3). Gelatinization temperature ranges for
CF and WGWF were 65.25-78.25 °C and 56.76-69.95 °C, respectively. Some
displacement in the peak temperatures, giving narrow peaks, was observed in the 50%
blend.

3.4.2. Thermal properties of extrudates

The effect of the extrusion variables and WGWF on thermal properties of
extrudates is shown in Table 3. There were no significant models that fit the DSC
parameters, with the exception of the positive quadratic effect of the extrusion
temperature on the conclusion temperature (T_c), and the linear negative effect of the
WGWF on the enthalpy. Certain polysaccharides such as fiber may restrict water
availability and consequently restrict gelatinization of starch resulting in a decrease in
enthalpy variation [45]. The T_p observed for the extrudates was around 104-114 °C and
the endothermic absorption ranged from 0.93-4.60 J/g (Table 2), which corresponds to
the melting of the amylose-lipid complex formed during extrusion [13]. Considering
that most of the starch was gelatinized during extrusion and that disruption of molecular
bonds must have occurred, the amylose released and available to bind lipids may have
led to the formation of more stable amylose-lipid complexes.

There were no endotherms around 50-80 °C (temperature range around which
gelatinization of cereal starch occurs) after extrusion cooking, indicating that all native
crystalline structures were transformed or that the starches were fully gelatinized. This
result is similar to that reported by [46], who observed no residual gelatinization
enthalpy for wholemeal products after extrusion at 110 °C.

4. Conclusions
Extrusion conditions (feed moisture and temperature) and formulation, such as that containing whole grain wheat flour, have great impact on starch transformation and extrudates structure. A detailed study of the raw materials and flour blends in association with extrusion conditions allows understanding the macromolecular transformations and the control of final product quality. WGWF and feed moisture induced the greatest effects on SME and WSI (measurements of extrusion process severity). There were changes in the crystallinity pattern of starch due to extrusion processing with melting of A-type (unprocessed flours and non-extruded flour blends) to form V-type (extrudates) starches. The DSC scanning confirmed that the extrusion process provided complete starch gelatinization, and extrudates only displayed a peak corresponding to the amylose-lipid complex. In conclusion, the starch nature (corn or wheat) as well as the presence of fiber governed starch transformations and interactions (gelatinization and amylose-lipid complex formation). Overall, the extrusion of whole grain wheat flour is a feasible process for which starch gelatinization, starch complex formation and related functional properties can be studied as a function of extrusion variables with helpful information to obtain extrudates with improved nutritional properties, such as breakfast cereals.

5. Acknowledgments

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The authors have declared no conflict of interest.

6. References


**Figure captions**

**Figure 1.** Response surface plots for water absorption index (WAI) and water solubility index (WSI) as a function of whole grain wheat flour (WGWF), feed moisture and temperature (A-F).

A- WAI as function of WGWF and temperature; B- WSI as function of WGWF and temperature; C- WAI as function of WGWF and moisture; D- WSI as function of WGWF and moisture; E- WAI as function of moisture and temperature; F- WSI as function of moisture and temperature.

**Figure 2.** X-ray diffraction patterns of unprocessed flour blends (corn flour and whole grain wheat flour) (G) and extruded cereals (A-F) (1-18 refer to the trials – see Table 2).

**Figure 3.** DSC profiles of unprocessed flours and blends composed of corn flour (CF) and whole grain wheat flour (WGWF) in excess water. P1 (peak 1), P2 (peak 2) and P3 (peak 3).
Figure 1
Figure 2

X axis: 2θ (degree). Y axis: normalized intensity.
Figure 3

*P1: peak 1 (wheat starch transition); P2: peak 2 (corn starch transition); P3: peak 3 (amylose-lipid complex transition). CF: corn flour. WGWF: whole grain wheat flour.
**Table 1.** Characteristics of the flours used as raw materials.

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<th>Whole grain wheat flour</th>
<th>Corn flour</th>
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<td>Moisture</td>
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<td>11.4±0.21</td>
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<td>Protein</td>
<td>12.7±0.16</td>
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<td>Fat</td>
<td>1.72±0.08</td>
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<tr>
<td>Ash</td>
<td>1.6±0.01</td>
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<td><strong>Carbohydrates (%)</strong></td>
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<td>Digestible starch</td>
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<td>Total starch</td>
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<td><strong>Particle size distribution (%)</strong></td>
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*Oliveira et al., 2015*
### Table 2. Central composite rotatable design for extruded products.

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<th>x₂</th>
<th>x₃</th>
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<th>Feed moisture (g/100g)</th>
<th>Temperature (°C)</th>
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<td>50</td>
<td>19</td>
<td>110</td>
<td>126.67±2.31</td>
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</table>

1The temperature refers to barrel temperature of zones 3 and 4 which was kept equal each other. The zones were counted from feeding to die (1-4). x₁: coded value for whole grain wheat flour ratio; x₂: coded value for feed moisture; x₃: coded value for temperature. 1Specific mechanical energy (Wh.kg⁻¹); 2water absorption index (g gel/g sample); 3water solubility index (%); 4onset temperature (°C); 5peak temperature (°C); 6offset or conclusion temperature (°C); 7enthalpy change (J/g); 8starch crystallinity (%).

*Values represent average triplicate determinations.
Table 3. Estimated coefficients (coded values based) for the responses (dependent variables).

<table>
<thead>
<tr>
<th>Factor</th>
<th>SME&lt;sup&gt;1&lt;/sup&gt;</th>
<th>WAI&lt;sup&gt;2&lt;/sup&gt;</th>
<th>WSI&lt;sup&gt;3&lt;/sup&gt;</th>
<th>T&lt;sub&gt;o&lt;/sub&gt;&lt;sup&gt;4&lt;/sup&gt;</th>
<th>T&lt;sub&gt;p&lt;/sub&gt;&lt;sup&gt;5&lt;/sup&gt;</th>
<th>T&lt;sub&gt;c&lt;/sub&gt;&lt;sup&gt;6&lt;/sup&gt;</th>
<th>ΔH&lt;sup&gt;7&lt;/sup&gt;</th>
<th>SC&lt;sup&gt;8&lt;/sup&gt;</th>
</tr>
</thead>
<tbody>
<tr>
<td>Constant</td>
<td>119.77&lt;sup&gt;*&lt;/sup&gt;</td>
<td>5.77</td>
<td>16.09</td>
<td>102.71</td>
<td>107.65</td>
<td>112.10</td>
<td>2.21</td>
<td>18.74</td>
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<tr>
<td>WGWF (x₁)</td>
<td>-12.76&lt;sup&gt;*&lt;/sup&gt;</td>
<td>-0.59&lt;sup&gt;*&lt;/sup&gt;</td>
<td>-3.17&lt;sup&gt;*&lt;/sup&gt;</td>
<td>ns</td>
<td>ns</td>
<td>ns</td>
<td>-0.49&lt;sup&gt;*&lt;/sup&gt;</td>
<td>ns</td>
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<tr>
<td>WGWF (x₁x₁)</td>
<td>1.67</td>
<td>0.26</td>
<td>-</td>
<td>ns</td>
<td>ns</td>
<td>ns</td>
<td>ns</td>
<td>ns</td>
</tr>
<tr>
<td>Moisture (x₂)</td>
<td>-13.30&lt;sup&gt;*&lt;/sup&gt;</td>
<td>0.04</td>
<td>-2.76&lt;sup&gt;*&lt;/sup&gt;</td>
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<td>Moisture (x₂x₂)</td>
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<td>0.42&lt;sup&gt;*&lt;/sup&gt;</td>
<td>1.85&lt;sup&gt;*&lt;/sup&gt;</td>
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<tr>
<td>Temperature (x₃)</td>
<td>-6.20</td>
<td>0.13</td>
<td>-</td>
<td>ns</td>
<td>ns</td>
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</tr>
<tr>
<td>Temperature (x₃x₃)</td>
<td>-6.47</td>
<td>0.37&lt;sup&gt;*&lt;/sup&gt;</td>
<td>1.46&lt;sup&gt;*&lt;/sup&gt;</td>
<td>ns</td>
<td>ns</td>
<td>2.22&lt;sup&gt;*&lt;/sup&gt;</td>
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<tr>
<td>WGWF x Moisture (x₁x₂)</td>
<td>-1.58</td>
<td>-0.03</td>
<td>-</td>
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<td>ns</td>
<td>ns</td>
<td>ns</td>
<td>2.70&lt;sup&gt;*&lt;/sup&gt;</td>
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<td>WGWF x Temperature (x₁x₃)</td>
<td>1.25</td>
<td>-0.17</td>
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<td>ns</td>
<td>ns</td>
<td>ns</td>
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<tr>
<td>Moisture x Temperature (x₂x₃)</td>
<td>-1.58</td>
<td>0.04</td>
<td>-</td>
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<td>ns</td>
<td>ns</td>
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<td>R² (%)</td>
<td>75.54</td>
<td>73.92</td>
<td>76.47</td>
<td>45.50</td>
<td>38.17</td>
<td>53.65</td>
<td>61.55</td>
<td>53.76</td>
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</table>

<sup>1</sup> Specific mechanical energy; <sup>2</sup> water absorption index; <sup>3</sup> water solubility index; <sup>4</sup> onset temperature; <sup>5</sup> peak temperature; <sup>6</sup> offset or conclusion temperature; <sup>7</sup> enthalpy change; <sup>8</sup> starch crystallinity.

* Statistically significant at P<0.10.

ns = not significant
<table>
<thead>
<tr>
<th>Unprocessed flours and blends</th>
<th>Wheat starch transition (peak 1)</th>
<th>Corn starch transition (peak 2)</th>
<th>Amylose-lipid complex transition (peak 3)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$T_o$ ($^\circ$C)</td>
<td>$T_p$ ($^\circ$C)</td>
<td>$T_c$ ($^\circ$C)</td>
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<td>CF 100</td>
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<td>WGWF 50</td>
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<td>62.57±0.32a</td>
<td>66.51±1.17a</td>
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<tr>
<td>WGWF 79.76</td>
<td>56.04±0.23a</td>
<td>61.96±0.36a</td>
<td>67.42±0.34a</td>
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</table>

*Values followed by the same letter in the same row, within the same peak, are not significantly different at $P<0.10$. CF 100: 100% of corn flour; WGWF: 100% of whole grain wheat flour; WGWF 20.24: 0.24% of whole grain wheat flour; WGWF 50: 50% of whole grain wheat flour; WGWF 79.76: 79.76% of whole grain wheat flour; $T_o$: onset temperature; $T_p$: peak temperature; $T_c$: offset or conclusion temperature; $\Delta H$: enthalpy change.