



The effect of vacuum frying on starch gelatinization and its *in vitro* digestibility in starch–gluten matrices



Ingrid Contardo^a, Javier Parada^b, Angel Leiva^c, Pedro Bouchon^{a,*}

^a Department of Chemical and Bioprocess Engineering, Pontificia Universidad Católica de Chile, P.O. Box 306, Santiago 6904411, Chile

^b Institute of Food Science and Technology, Universidad Austral de Chile, P.O. Box 47, Valdivia, Chile

^c Department of Physical Chemistry, Pontificia Universidad Católica de Chile, P.O. 360, Santiago 6904411, Chile

ARTICLE INFO

Article history:

Received 8 May 2015

Received in revised form 14 September 2015

Accepted 7 October 2015

Available online 8 October 2015

Keywords:

Frying

Vacuum frying

Starch

Gelatinization

In vitro digestibility

Starch digestibility

ABSTRACT

Starch digestibility in a food matrix depends on processing conditions that may affect its physical state and microstructure. Starch gelatinization is one critical change that takes place during frying which could be affected during low-pressure processing. This study assessed the effect of vacuum frying on starch gelatinization and its *in vitro* digestibility. Laminated dough was made of a reconstituted blend of wheat starch (88% d.b.) and gluten (12% d.b.). Samples were fried under vacuum (6.5 kPa, $T_{\text{water-boiling-point}} = 38^\circ\text{C}$) or atmospheric conditions up to bubble-end point, maintaining a thermal driving force of 70°C ($T_{\text{oil}} - T_{\text{water-boiling-point}} = 70^\circ\text{C}$). Vacuum fried samples showed less starch gelatinization (28%), less rapidly available glucose (27%), and more unavailable glucose (70%) than their atmospheric counterparts (which presented 99% starch gelatinization, 40% rapidly available glucose, and 46% unavailable glucose), and the values were close to those of raw dough. These results show how vacuum processing may be used to control the degree of starch gelatinization and related digestibility.

© 2015 Elsevier Ltd. All rights reserved.

1. Introduction

Starch is the main carbohydrate in human nutrition. It is mostly found in corn, potatoes, wheat, cassava, and rice, and it is used in foods as a thickening, gelling, and structure-forming agent (Bertolini, 2010). Most of these properties are triggered when starch is heated in the presence of liquid water (Biliaderis, 1991). Under these circumstances, starch granules swell and lose their crystallinity and molecular organization in a process known as gelatinization (Wang & Copeland, 2013).

The temperature range for gelatinization and the energy required for that process depend on the botanical source, and they are often characterized through differential scanning calorimetry (DSC) (Gonera & Cornillon, 2002). The amount of water available is also a critical factor (Baks, Ngene, van Soest, Janssen, & Boom, 2007). In fact, at intermediate concentrations of water (30–70% w/w), the gelatinization temperature range may be extended (Biliaderis, 2009; Parker & Ring, 2001). Furthermore, the addition of sugars such as sucrose, glucose, and fructose to solutions containing starch may delay the process due to water accessibility limitations (Molina, Leiva, & Bouchon, 2015; Mason, 2009; Sopade,

Halley, & Junming, 2004). An increase in the heating rate may increase the onset gelatinization temperature (Ovalle, Cortés, & Bouchon, 2013), whereas freezing prior to heating may delay it (Molina et al., 2015).

When starch granules are gelatinized, the disruption of their structure increases their susceptibility to enzymatic degradation and related digestibility (Holm, Lundquist, Björck, Eliasson, & Asp, 1988). Also, when starch granules are trapped within a matrix (as is the case in dough) differences in starch digestibility may be associated with the changes in the physical state of the granule itself as well as the type of microstructure developed during processing. This may hinder the accessibility of hydrolytic enzymes during digestion (Lee, Kim, Choi, & Moon, 2012; Parada & Aguilera, 2011a; Singh, Dartois, & Kaur, 2010). Thus, structural changes which occur during thermal food processing may change the postprandial response (Bravo, Siddhuraju, & Saura-Calixto, 1998; Englyst, Englyst, Hudson, Cole, & Cummings, 1999; Kawai, Matsusaki, Hando, & Hagura, 2013; Lee et al., 2012; Parada & Aguilera, 2011a,b). In order to account for such differences, Englyst et al. (1999) developed an *in vitro* enzymatic method of classifying starch based on its digestibility. The approach involves measuring the amount of glucose released from a food during timed incubation under standardized conditions. Rapidly available glucose (RAG) was defined as the fraction that was obtained after 20 min of hydrolysis. Slowly available glucose (SAG) was said to

* Corresponding author at: 4860 Vicuña Mackenna Ave. P.O. Box 306, Santiago 6904411, Chile.

E-mail address: pbouchon@ing.puc.cl (P. Bouchon).

be the fraction obtained between 20 and 120 min of hydrolysis. These amounts of glucose are likely to be available for rapid and slow absorption, respectively, in the human small intestine. Finally, unavailable glucose (UG) was defined as the fraction that could not be released after 120 min of hydrolysis.

Deep-fat frying involves immersing foods in edible oils and fats at elevated temperatures in order to induce rapid dehydration and related microstructural changes (Moreno, Brown, & Bouchon, 2010). In starchy matrices, starch gelatinization is one of these critical changes. Excess consumption of fat, a main component of fried food, and the formation of toxic compounds within the food (e.g. acrylamide) have led the food industry to develop new alternatives such as vacuum frying (Dueik & Bouchon, 2011). This process is carried out under pressures well below atmospheric levels, reducing the boiling point of water, which makes it possible to substantially reduce the frying temperature (Garayo & Moreira, 2002). These processing conditions allow for better preservation of nutrients, minimize oil degradation, and may reduce oil absorption while maintaining the organoleptic properties of fried fruits and vegetables (Da Silva & Moreira, 2008; Dueik, Robert, & Bouchon, 2010; Fan, Zhang, Xiao, Sun, & Tao, 2005). Interestingly, starch gelatinization may be impaired under vacuum conditions, as determined by Ovalle et al. (2013) using *in situ* vacuum hot-stage microscopy. Similar results were recently reported by Oginni, Sobukola, Henshaw, Afolabi, and Munoz (2015) when vacuum frying a cassava gluten-based snack. These processing conditions may in turn affect starch digestibility.

The objective of this study was to assess the effect of vacuum conditions on starch gelatinization and *in vitro* digestibility when a restructured matrix made with native wheat gluten and starch was fried. In accordance, this research aims to understand how starch digestibility can be modulated when processing under vacuum conditions in order to illustrate how processing may help tune nutritional properties.

2. Materials and methods

2.1. Materials

Starch-gluten matrices (laminated dough) were prepared using native wheat starch (Comercial Venser S.A., Santiago, Chile), vital wheat gluten (Asitec S.A., Santiago, Chile), and distilled water. High oleic sunflower oil (Camilo Ferrón Chile S.A., Santiago, Chile) was used as the frying medium in all experiments.

Pepsin-P7000, amylglucosidase-A7095, pancreatin-7545 (Sigma–Aldrich, St Louis, MO, USA), and invertase-390203D (VWR International Ltd., Poole, UK) were used in *in vitro* digestibility essays.

2.2. Sample preparation

Dough was prepared following the procedure described by Moreno et al. (2010) with a few modifications. Formulations were prepared using native wheat starch (88% d.b.) and wheat gluten (12% d.b.) and steps were taken to ensure that all of the dough had the same final moisture content (40% w.b.). The amount of

water added to the dry ingredients was a function of the initial water content of the dry ingredients and was adjusted in order to ensure that all samples contained the specified amount. Table 1 shows the initial moisture content of the dry ingredients and the dough.

To form the dough, the dry ingredients were first mixed for 3 min using a 5K5SS mixer (Kitchen Aid, St. Joseph, MI, USA) equipped with a K5AB flat beater at 40 rpm. Half of the water was added at 15 °C while mixing for 2 min. The remaining amount was added at 85 °C while mixing for 2 min. The dough was then allowed to rest for 40 min inside of a plastic film. Next, the dough was sheeted using a LSB516 dough sheeter (Doyon, Saint-Côme-Linière, Quebec, Canada), obtaining a final thickness of 2 mm. The sheeted dough was cut into squares ($3.8 \times 3.8 \text{ cm}^2$) with constant weight ($3.56 \pm 0.10 \text{ g}$). The samples were stored in plastic film to prevent dehydration.

2.3. Frying experiments under vacuum or atmospheric conditions

Frying experiments were performed using an electrically heated 10 l stainless steel fryer that could be hermetically covered with a stainless steel lid. The fryer was filled with 3.5 l high-oleic sunflower oil. The container was thermostatically controlled to maintain the set frying temperature ($\pm 2 \text{ }^\circ\text{C}$), as described by Dueik et al. (2010). The fryer basket rod was connected to a rotor that was used to stir the oil at 40 rpm before frying in order to minimize temperature gradients. An equivalent thermal driving force of 70 °C was used to compare vacuum and atmospheric frying. The thermal driving force was defined by Mariscal and Bouchon (2008) as the difference between the oil temperature and the boiling temperature of water at the working pressure. Those temperatures are 100 °C under atmospheric conditions and 38 °C under the vacuum conditions used in this study (6.5 kPa). This yielded frying temperatures of 170 and 108 °C, respectively. The fryer was covered during both sets of experiments. Once the oil reached the required frying temperature, 8 slices ($\sim 28 \text{ g}$) of dough were placed in the frying basket in order to minimize the drop in temperature. The slices were covered with a grid in order to prevent them from floating. In vacuum frying, the slices were loaded and the vessel was depressurized (in $\approx 20 \text{ s}$). Once the vessel reached the target pressure, the basket was dipped into the frying oil for the required period of time.

Two frying times were used at each pressure: the time required to reach bubble-end point ($\approx 2.5\%$ moisture), which is defined as t_{ep} , and half this time ($t_{hp} = t_{ep}/2$). During atmospheric frying, t_{ep} and t_{hp} were 180 and 90 s. During vacuum frying, the frying times were 120 and 60 s, respectively. The samples were then removed from the fryer and stored in a desiccator for further analyses. Vacuum fried samples were removed from the oil before the vessel was pressurized. In addition, some batches were centrifuged at 400 rpm for 150 s just after frying. In vacuum frying, samples were centrifuged after they were removed from the oil but before the vessel was pressurized.

2.4. Analytical methods

2.4.1. Oil content

Total oil content of grounded samples was determined gravimetrically by Soxhlet extraction using petroleum ether (official method 920.39; AOAC, 1995).

2.4.2. Moisture content

The oil-free samples were dried in a forced oven at 105 °C to constant weight (official method 945.15; AOAC, 1995). Moisture content was determined gravimetrically by weight difference.

Table 1

Initial moisture content of the dry ingredients and the laminated dough (unprocessed matrix). Data are means $\pm$ standard deviation ($n = 6$).

	Moisture content (g water/100 g dry solids)
Wheat starch	12.43 $\pm$ 0.92
Gluten	5.73 $\pm$ 0.26
Dough	61.12 $\pm$ 0.41

2.4.3. Texture analysis

The texture of the samples was measured using a three-point bending test following the procedure described by Dueik et al. (2010). The test was carried out in a TA.XT2 Texture Analyzer (Texture Technologies Corp., Scarsdale, NY, USA). Each sample was placed on two parallel edges (support span of 16 mm) in order to apply the load centrally. A 2.5 mm-thick steel blade with a flat edge was used to fracture the sample at a speed of 10 mm/s. Texture measurements of the samples were conducted at room temperature (20 °C). The maximum breaking force ($F_{\max}$) at the fracture point was obtained using Texture Expert software version 1.16.

2.5. Degree of starch gelatinization

The degree of starch gelatinization was determined using a Mettler Toledo 821 DSC (Mettler-Toledo Inc., Schwerzenbach, Switzerland) based on the procedure described by Kawa and Moreira (2001). Prior to the DSC measurements, the unprocessed samples were dehydrated in an oven at 40 °C for at least 24 h. All samples were then ground up and sieved using a 60 mesh sieve. Four mg of sample was placed in a DSC aluminium pan (Part No. 03190029, Perkin Elmer, Norwalk, CT, USA) and distilled water was added to yield a water to sample ratio of 4:1. It was then hermetically sealed and kept in room temperature for ~12 h. The samples were scanned from 35 to 90 °C at a heating rate of 10 °C/min. Perkin Elmer Pyris Version 3.50 software was used to control the experimental conditions. The onset (T_o), peak (T_p), and endset (T_e) transition temperatures and gelatinization enthalpy (ΔH) were computed. The degree of starch gelatinization (DG) was determined using Eq. (1):

$$DG (\%) = \left(\frac{\Delta H_{\text{raw}} - \Delta H_{\text{fried}}}{\Delta H_{\text{raw}}} \right) \times 100 \quad (1)$$

2.6. In vitro starch digestibility

In vitro starch digestibility was determined according to Englyst et al. (1999) with some variations. Digestion was performed in three stages.

Stage 1: About 1.5 g of each sample was placed in polypropylene tubes (50 cm³) with 5 ml of 50% saturated benzoic solution acid and 10 ml of freshly prepared pepsin guar-gum solution (5 g pepsin/ml and 5 g guar gum/ml in 0.05 M HCl). Each tube was covered, vigorously vortex-mixed, and placed in a water bath at 37 °C for 30 min to allow for protein hydrolysis. Next, 5 ml of 0.5 M sodium acetate buffer solution (pH 5.2) and five glass balls (15 mm diameter) were added to each tube, shaken gently, and kept in the water bath for 3 min to equilibrate.

Stage 2: Next, 5 ml of fresh enzyme mixture of pancreatine–amylloglucosidase–invertase (18 g, 4 ml and 6 ml, respectively, per 100 ml enzyme mixture) was added to each tube. The tubes were covered, gently mixed, and placed horizontally in the 37 °C water bath. Shaking (137 rpm) was immediately started. Each tube was removed from the bath exactly 20 and 120 min after the enzyme mixture was added; 0.2 ml of the contents was added to 4 ml absolute ethanol and vortex mixed to stop the hydrolysis. These corresponded to the G_{20} (glucose concentration at 20 min) and G_{120} (glucose concentration at 120 min) portions. Each tube was covered, vortex mixed vigorously, and cooled in an ice water bath for 15 min.

Stage 3 (extreme gelatinization and digestion): Ten ml of 7 M potassium hydroxide solution was added and mixed. The tubes were placed horizontally in an ice water bath and shaken for 30 min. Then, 0.2 ml of the content was added to 1 ml acetic acid

1 M solution and 40 μ l of amylloglucosidase solution were added (1:7 dilution). The tubes were covered, vigorously vortex mixed, and placed in a water bath at 70 °C for 30 min. They were cooled in a water ice bath for 15 min, and allowed to reach room temperature before 12 ml of absolute ethanol was added. This tube corresponded to the TG (total glucose) portion.

Glucose concentration in the G_{20} , G_{120} , and TG portions were measured using glucose oxidase and peroxidase assay kit GAGO-20 (Sigma–Aldrich, St Louis, MO). Rapidly available glucose (RAG), slowly available glucose (SAG), and unavailable glucose (UG) fractions were determined as follows:

$$RAG (g/100_g) = G_{20}/TG \times 100 \quad (2)$$

$$SAG (g/100_g) = (G_{120} - G_{20})/TG \times 100 \quad (3)$$

$$UG (g/100_g) = (TG - G_{120})/TG \times 100 \quad (4)$$

2.7. Statistical analysis

The reported results correspond to the arithmetic mean of three batches $\pm$ standard deviation unless otherwise specified. Experimental data were tested by analysis of one-way variance (ANOVA) and means separation was achieved using the LSD method at 95% confidence, with Statgraphics plus 5.1 (Manugistics, Inc., Rockville, MD, USA).

3. Results and discussion

3.1. Oil uptake of starch–gluten matrices during frying and vacuum frying

Table 2 shows the oil uptake of the laminated dough after frying up to t_{ep} (bubble-end point) and t_{hp} ($=t_{ep}/2$), using a thermal driving force of $\Delta T = 70$ °C, under vacuum (6.5 kPa) or atmospheric conditions. Vacuum frying significantly increased the oil absorption of the samples after frying compared to atmospheric frying. This may be related to the development of a weaker structure during oil immersion which could be more vulnerable to oil infiltration during vacuum frying. This behavior also may be linked to the fast dehydration and shorter frying times needed to achieve bubble-end point during vacuum frying.

Centrifugation proved to be an effective post-processing step for reducing oil absorption after frying and vacuum frying. Similar results have been obtained by other authors when vacuum frying fruits and vegetables, such as potatoes, carrots, and apples (Da Silva & Moreira, 2008; Dueik, Moreno, & Bouchon, 2012).

Interestingly, we found higher oil reduction in vacuum fried laminated dough after centrifugation (40 and 48% reduction when frying for t_{hp} and t_{ep} , respectively) compared to atmospheric fried

Table 2

Oil uptake of the laminated dough after frying up to t_{ep} (bubble-end point) and t_{hp} ($=t_{ep}/2$), using a thermal driving force of 70 °C ($T_{oil} - T_{water-boiling-point} = 70$ °C), under vacuum (6.5 kPa) or atmospheric conditions. Data are means $\pm$ standard deviation ($n = 3$).

	Frying time	Oil content after frying (g oil/g de-fatted dry solids)	Oil content after frying and centrifuging (g oil/g de-fatted dry solids)
Vacuum frying	$t_{hp} = 60$ s	0.417 ± 0.010^a	0.234 ± 0.011^e
	$t_{ep} = 120$ s	0.487 ± 0.007^b	0.256 ± 0.007^f
Atmospheric frying	$t_{hp} = 90$ s	0.367 ± 0.008^c	0.277 ± 0.011^g
	$t_{ep} = 180$ s	0.435 ± 0.009^d	0.342 ± 0.008^h

Different superscripts denote significant differences ($p < 0.05$).

samples (25% and 21% reduction when frying for t_{hp} and t_{ep} , respectively). Their final oil content was actually significantly lower ($p < 0.05$) than that of atmospheric fried samples after centrifugation. This could be linked to the formation of a frailer structure during vacuum frying, which could make the samples more susceptible to oil removal during centrifugation.

3.2. Texture changes of starch–gluten matrices during frying and vacuum frying

In order to better understand the structural changes that take place during frying, the texture of the fried slices was analyzed. Hardness, which corresponds to the maximal force produced upon compression of the sample, was used as a texture descriptor.

Fig. 1 shows changes in hardness when frying for t_{hp} or t_{ep} under vacuum or under atmospheric conditions. The maximum force required to break the fried matrices was significantly reduced ($p < 0.05$) when the frying pressure was decreased after both frying times. During frying, the operating pressure defines the boiling point of water, which is the temperature of the evaporation front.

As frying progresses, the front moves toward the interior of the product and a dehydrated crust is formed (i.e. no liquid water is left). The temperature of that crust may rise above the water boiling-point (Bouchon & Pyle, 2005). Conversely, the temperature inside of the food (core region), which still contains liquid water, is restricted to somewhere around the boiling point (38 °C in the case of vacuum frying at 6.5 kPa). Increased hardness is a result of the development of the dehydrated crust, which includes the entire product in the case of thin laminated dough fried up to bubble-end point (Bouchon & Aguilera, 2001). Starch gelatinization is a key process that requires both the presence of liquid water and adequate heating to reach temperatures between 55 and 75 °C depending on the system and the nature of the starch (Ovalle et al., 2013). A low pressure (6.5 kPa) and low water boiling-point (38 °C) may impair the process because these conditions make it difficult to meet both requirements simultaneously. As a result, the hardness of the structure is reduced, as shown in the Figure. This behavior is consistent with results presented by Sobukola, Dueik, and Bouchon (2013) when vacuum frying using different pressure conditions, and may help explaining the higher oil content of vacuum fried matrices as well as the higher efficiency of post-centrifugation after vacuum frying compared to atmospheric frying.

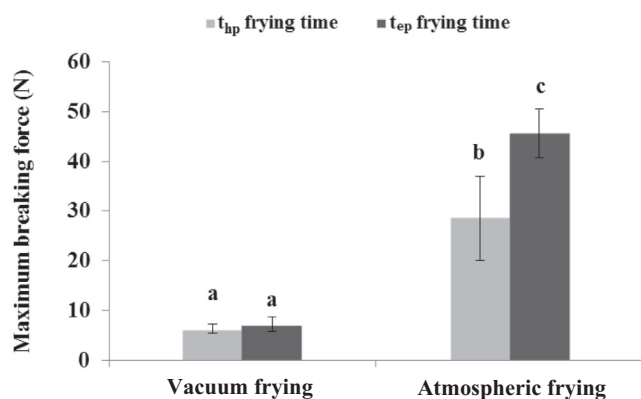


Fig. 1. Maximum breaking force of vacuum fried (6.5 kPa) and atmospheric fried laminated dough, after frying up to bubble-end point (t_{ep}) or half this time ($t_{hp} = t_{ep}/2$), using a thermal driving force of 70 °C ($T_{oil} - T_{water-boiling-point} = 70$ °C). Data are means $\pm$ standard deviation ($n = 12$). Different superscripts denote significant differences ($p < 0.05$).

3.3. Degree of starch gelatinization during frying

In order to assess the importance of starch gelatinization in structure formation, the gelatinization degree was analyzed. Fig. 2 shows the results of the matrices fried under the different conditions. The gelatinization degree was significantly lower ($p < 0.05$) in vacuum fried matrices compared to atmospheric fried ones. The mean values in vacuum fried samples were 8.90% and 27.47%, and those found in atmospheric fried samples were 96.60% and 98.61% after frying for t_{hp} or t_{ep} , respectively. These results are consistent with those reported by Ravli, Da Silva, and Moreira (2013) for vacuum frying sweet potatoes. Interestingly, no significant differences in F_{max} were found between vacuum fried samples after t_{hp} and t_{ep} . However, a slight increase in starch gelatinization was found which was probably not big enough to increase hardness. Conversely, a significant ($p < 0.05$) increase in F_{max} was observed in atmospheric fried samples. No significant differences in starch gelatinization were observed when comparing samples fried during t_{hp} or t_{ep} . The increase in hardness could be attributed to further dehydration of the matrix (Bouchon & Aguilera, 2001; Gazmuri & Bouchon, 2009).

Table 3 presents the onset (T_o), peak (T_p), endset (T_e) transition temperatures, the gelatinization temperature range ($T_e - T_o$), and the gelatinization enthalpy (ΔH) after vacuum and atmospheric frying. The transition temperatures of the ungelatinized starch granules were not really affected by the frying conditions and did not differ significantly from those found in the raw dough. The gelatinization enthalpies of the starch granules present in atmospheric fried matrices were significantly lower ($p < 0.05$) than those obtained in vacuum fried ones, and these were very close to those found in the raw dough. Overall, these results highlight the importance of starch gelatinization in structure formation as well as the lack of structure in vacuum fried matrices.

3.4. In vitro digestibility of starch in gluten-starch matrices

Starch digestibility may depend on the physical state of starch as well as factors such as the state of the matrix developed during processing. Fig. 3 shows the RAG (rapidly available glucose), SAG

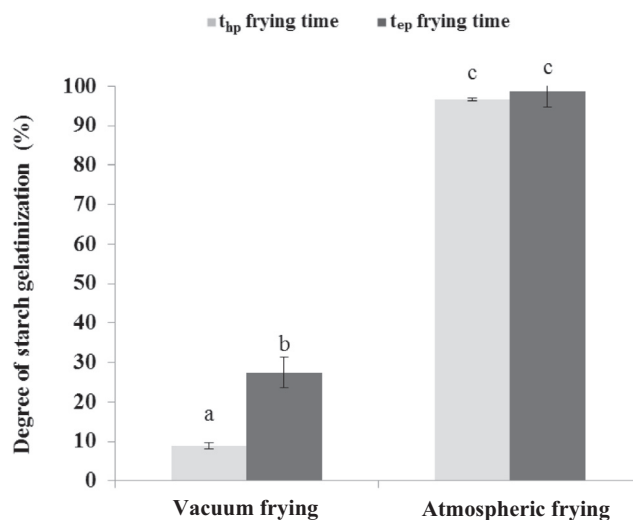


Fig. 2. Degree of starch gelatinization of vacuum fried (6.5 kPa) and atmospheric fried laminated dough, after frying up to bubble-end point (t_{ep}) or half this time ($t_{hp} = t_{ep}/2$), using a thermal driving force of 70 °C ($T_{oil} - T_{water-boiling-point} = 70$ °C). Data are means $\pm$ standard deviation ($n = 3$). Different superscripts denote significant differences ($p < 0.05$).

Table 3

Onset (T_o), peak (T_p), endset (T_e) transition temperatures, gelatinization temperature range ($T_e - T_o$), and gelatinization enthalpy (ΔH) of laminated dough after frying up to t_{ep} (bubble-end point) and $t_{hp}(=t_{ep}/2)$, using a thermal driving force of 70 °C ($T_{oil} - T_{water-boiling-point} = 70$ °C), under vacuum (6.5 kPa) or atmospheric conditions. Data are means $\pm$ standard deviation ($n = 3$).

	T_o (°C)	T_p (°C)	T_e (°C)	$T_e - T_o$ (°C)	ΔH (J/g)
Dough (raw)	56.99 $\pm$ 0.78 ^a	63.25 $\pm$ 0.29 ^{ab}	67.40 $\pm$ 2.39 ^a	10.41 $\pm$ 1.78 ^a	6.370 $\pm$ 1.32 ^a
Vacuum frying (t_{hp})	57.95 $\pm$ 1.30 ^{ab}	62.19 $\pm$ 1.57 ^a	66.33 $\pm$ 3.51 ^a	8.38 $\pm$ 4.65 ^a	5.803 $\pm$ 0.13 ^a
Vacuum frying (t_{ep})	58.00 $\pm$ 1.86 ^{ab}	63.12 $\pm$ 0.78 ^{ab}	68.22 $\pm$ 0.45 ^a	10.23 $\pm$ 1.41 ^a	4.620 $\pm$ 0.25 ^b
Atmospheric frying (t_{hp})	60.52 $\pm$ 2.05 ^b	65.28 $\pm$ 2.82 ^b	69.75 $\pm$ 3.46 ^a	9.23 $\pm$ 2.41 ^a	0.217 $\pm$ 0.05 ^c
Atmospheric frying (t_{ep})	59.29 $\pm$ 1.28 ^{ab}	63.36 $\pm$ 0.50 ^{ab}	69.07 $\pm$ 1.80 ^a	9.78 $\pm$ 3.03 ^a	0.089 $\pm$ 0.02 ^c

Different superscripts in each column denote significant differences ($p < 0.05$).

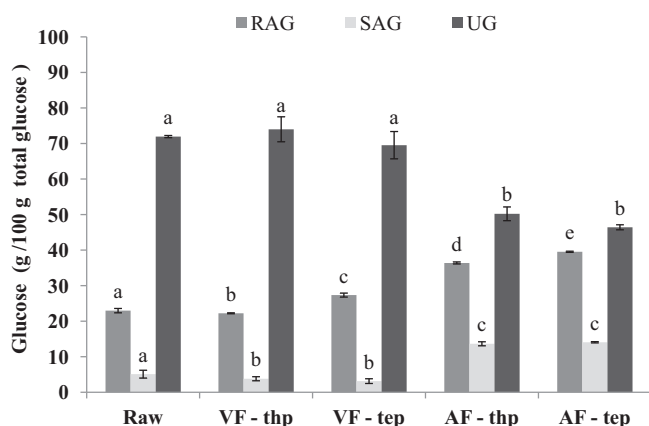


Fig. 3. RAG (rapidly available glucose), SAG (slowly available glucose), and UG (unavailable glucose) of vacuum fried (VF, 6.5 kPa) and atmospheric fried (AF) laminated dough, after frying up to bubble-end point (t_{ep}) or half this time ($t_{hp} = t_{ep}/2$), using a thermal driving force of 70 °C ($T_{oil} - T_{water-boiling-point} = 70$ °C). Data are means $\pm$ standard deviation ($n = 3$). Values in the same series (same colour) with different superscripts denote significant differences ($p < 0.05$).

(slowly available glucose), and UG (unavailable glucose) fractions of vacuum and atmospheric fried matrices, as well as those found in raw samples.

The frying pressure had a significant effect on all fractions and similar behavior was observed in raw and vacuum fried matrices overall. RAG values in vacuum fried samples (22.21 and 27.35 g/100 g after frying for t_{hp} or t_{ep} , respectively) were very close to those obtained in unprocessed samples (22.96 g/100 g). These, in turn were significantly lower than those found in samples fried under atmospheric conditions (36.38 and 39.53 g/100 g, respectively). SAG values of vacuum fried samples were slightly lower (3.79 and 3.13 g/100 g after frying for t_{hp} or t_{ep} , respectively) than that of raw dough (5.09 g/100 g), while those of fried samples under atmospheric conditions were higher. Again, the UG values of vacuum fried samples were similar to those found in raw samples while the UG values of atmospheric fried samples were significantly lower. As explained earlier, ungelatinized starch is barely digestible (Parada & Aguilera, 2011a,b). Our results confirm this relationship along with the possibility that low pressure processing offers to control starch digestibility.

Interestingly, a higher SAG/RAG ratio was found in atmospheric fried samples. This may be due to the dense structure generated during heating, which increases hardness and delays the digestion rate (see Fig. 1). However, starch was hardly digested in vacuum-fried matrices even though they had a weak structure. The degree of starch gelatinization appears to have a dominant effect on the extent of starch digestion, whereas the state of the matrix could be linked to the rate of starch digestion.

4. Conclusion

The results showed that it is possible to reduce the degree of starch gelatinization in a starch-gluten laminated matrix during frying when using a vacuum level that defines a water boiling point that is lower to the one required for starch gelatinization. These operating conditions produce a weak structure that is more permeable to oil absorption, which in turn can facilitate surface oil removal during post-frying centrifugation.

In addition, vacuum frying allowed researchers to obtain a formulated matrix with levels of rapidly available glucose (RAG), slowly available glucose (SAG) and unavailable glucose (UG) fractions comparable to those obtained in raw dough. This is probably due to the low level of starch gelatinization. An important decrease in UG was obtained in samples fried under atmospheric conditions, in which, conversely, most of the starch was gelatinized.

Overall, this study shows how low-pressure processing can be used to control the degree of starch gelatinization in a starchy matrix and its physical and nutritional properties.

Acknowledgements

The authors would like to acknowledge the financial support of CONICYT – Chile through the Human Capital Formation Advanced Program National Doctoral Fellowship 2013 (Folio 21130468), Anillo Project ACT1105/12: Healthy food matrix design and the School of Engineering from Pontificia Universidad Católica de Chile.

References

- AOAC. (1995). *Official methods of analysis* (16th edition). Washington: Association of Official Analytical Chemists.
- Baks, T., Ngene, I. S., van Soest, J. J. G., Janssen, A. E. M., & Boom, R. M. (2007). Comparison of methods to determine the degree of gelatinisation for both high and low starch concentrations. *Carbohydrate Polymers*, 67(4), 481–490.
- Bertolini, A. C. (2010). Trends in starch applications. In A. C. Bertolini (Ed.), *Starches: Characterization, Properties and Applications* (First edition, pp. 1–20). Florida, EE.UU.: CRC Press.
- Biliaderis, C. G. (1991). The structure and interactions of starch with food constituents. *Canadian Journal of Physiology and Pharmacology*, 69(1), 60–78.
- Biliaderis, C. G. (2009). Structural transitions and related physical properties of starch. In J. BeMiller & R. Whistler (Eds.), *Starch: Chemistry and Technology* (Second Ed., pp. 293–372). EE.UU.: Academic Press.
- Bouchon, P., & Aguilera, J. M. (2001). Microstructural analysis of frying potatoes. *International Journal of Food Science & Technology*, 36, 669–676.
- Bouchon, P., & Pyle, D. L. (2005). Modelling oil absorption during post-frying cooling-II: Solution of the mathematical model, model testing and simulations. *Food and Bioprocess Processing*, 83(C4), 253–260.
- Bravo, L., Siddhuraju, P., & Saura-Calixto, F. (1998). Effect of various processing methods on the in vitro starch digestibility and resistant starch content of indian pulses. *Journal of Agricultural and Food Chemistry*, 46(11), 4667–4674.
- Da Silva, P. F., & Moreira, R. G. (2008). Vacuum frying of high-quality fruit and vegetable-based snacks. *LWT – Food Science and Technology*, 41(10), 1758–1767.
- Dueik, V., & Bouchon, P. (2011). Vacuum frying as a route to produce novel snacks with desired quality attributes according to new health trends. *Journal of Food Science*, 76(2), E188–95.
- Dueik, V., Moreno, M. C., & Bouchon, P. (2012). Microstructural approach to understand oil absorption during vacuum and atmospheric frying. *Journal of Food Engineering*, 111(3), 528–536.

- Dueik, V., Robert, P., & Bouchon, P. (2010). Vacuum frying reduces oil uptake and improves the quality parameters of carrot crisps. *Food Chemistry*, 119(3), 1143–1149.
- Englyst, K. N., Englyst, H. N., Hudson, G. J., Cole, T. J., & Cummings, J. H. (1999). Rapidly available glucose in foods: an in vitro measurement that reflects the glycemic response. *The American Journal of Clinical Nutrition*, 69(3), 448–454.
- Fan, L., Zhang, M., Xiao, G., Sun, J., & Tao, Q. (2005). The optimization of vacuum frying to dehydrate carrot chips. *International Journal of Food Science & Technology*, 40(9), 911–919.
- Garayo, J., & Moreira, R. (2002). Vacuum frying of potato chips. *Journal of Food Engineering*, 55, 181–191.
- Gazmuri, A. M., & Bouchon, P. (2009). Analysis of wheat gluten and starch matrices during deep-fat frying. *Food Chemistry*, 115(3), 999–1005.
- Gonera, A., & Cornillon, P. (2002). Gelatinization of starch/gum/sugar systems studied by using DSC, NMR, and CSLM. *Starch – Stärke*, 54, 508–516.
- Holm, J., Lundquist, J., Björck, I. M. E., Eliasson, A.-C., & Asp, N.-G. (1988). Degree in vitro, of starch gelatinization, and metabolic response in rats. *The American Journal of Clinical Nutrition*, 47, 1010–1016.
- Kawai, K., Matsusaki, K., Hando, K., & Hagura, Y. (2013). Temperature-dependent quality characteristics of pre-dehydrated cookies: structure, browning, texture, in vitro starch digestibility, and the effect on blood glucose levels in mice. *Food Chemistry*, 141(1), 223–228.
- Kawas, M. L., & Moreira, M. G. (2001). Effect of degree of starch gelatinization on quality attributes of fried tortilla chips. *Food Engineering and Physical Properties*, 66(2), 300–306.
- Lee, C. J., Kim, Y., Choi, S. J., & Moon, T. W. (2012). Slowly digestible starch from heat-moisture treated waxy potato starch: Preparation, structural characteristics, and glucose response in mice. *Food Chemistry*, 133(4), 1222–1229.
- Mariscal, M., & Bouchon, P. (2008). Comparison between atmospheric and vacuum frying of apple slices. *Food Chemistry*, 107(4), 1561–1569.
- Molina, M.T., Leiva, A., & Bouchon, P. (2015). Understanding the effect of freezing on starch microstructural changes during heating at high rates using in situ hot-stage video-microscopy and differential scanning calorimetry, (unpublished data).
- Mason, W. (2009). Starch: Chemistry and Technology. In J. BeMiller & R. Whistler (Eds.), *Starch Use in Food* (Second Ed., pp. 745–796). EE.UU.: Academic Press.
- Moreno, M. C., Brown, C., & Bouchon, P. (2010). Effect of food surface roughness on oil uptake by deep-fat fried products. *Journal of Food Engineering*, 101(2), 179–186.
- Oginni, O. C., Sobukola, O. P., Henshaw, F. O., Afolabi, W. A. O., & Munoz, L. (2015). Effect of starch gelatinization and vacuum frying conditions on structure development and associated quality attributes of cassava-gluten based snack. *Food Structure*, 3, 12–20.
- Ovalle, N., Cortés, P., & Bouchon, P. (2013). Understanding microstructural changes of starch during atmospheric and vacuum heating in water and oil through online in situ vacuum hot-stage microscopy. *Innovative Food Science & Emerging Technologies*, 17, 135–143.
- Parada, J., & Aguilera, J. M. (2011a). Microstructure, mechanical properties, and starch digestibility of a cooked dough made with potato starch and wheat gluten. *LWT – Food Science and Technology*, 44(8), 1739–1744.
- Parada, J., & Aguilera, J. M. (2011b). Review: starch matrices and the glycemic response. *Food Science and Technology International*, 17(3), 187–204.
- Parker, R., & Ring, S. G. (2001). Aspects of the physical chemistry of starch. *Journal of Cereal Science*, 34(1), 1–17.
- Ravli, Y., Da Silva, P., & Moreira, R. G. (2013). Two-stage frying process for high-quality sweet-potato chips. *Journal of Food Engineering*, 118(1), 31–40.
- Singh, J., Dartois, A., & Kaur, L. (2010). Starch digestibility in food matrix: a review. *Trends in Food Science & Technology*, 21(4), 168–180.
- Sobukola, O. P., Dueik, V., & Bouchon, P. (2013). Understanding the effect of vacuum level in structure development and oil absorption in vacuum-fried wheat starch and gluten-based snacks. *Food and Bioprocess Technology*, 6, 2010–2017.
- Sopade, P., Halley, P. J., & Junming, L. L. (2004). Gelatinisation of starch in mixtures of sugars. II. Application of differential scanning calorimetry. *Carbohydrate Polymers*, 58, 311–321.
- Wang, S., & Copeland, L. (2013). Molecular disassembly of starch granules during gelatinization and its effect on starch digestibility: a review. *Food & Function*, 4, 1564–1580.