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Effects of freezing, freeze drying and convective drying on *in vitro* gastric digestion of apples

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ABSTRACT

The influence of processing (freezing at −196 °C in liquid N_2, FN sample; freeze-drying at −50 °C and 30 Pa, FD sample; and convective drying at 60 °C and 2 m/s, CD sample) on apple (var. *Granny Smith*) behavior during *in vitro* gastric digestion was investigated. Dried apples (FD and CD samples) were rehydrated prior to digestion. Changes in carbohydrate composition, moisture, soluble solids, acidity, total polyphenol content (TPC), and antioxidant activity (AA) of apple samples were measured at different times during digestion. Processing resulted in disruption of the cellular structure during digestion, as observed by scanning electron microscopy, light microscopy, and changes in carbohydrate composition. Moisture content increased (6–11% dm_o), while soluble solids (55–78% dm_o), acidity (44–72% dm_o), total polyphenol content (30–61% dm_o), and antioxidant activity (41–87%) decreased in all samples after digestion. Mathematical models (Weibull and exponential models) were used to better evaluate the influence of processing on apple behavior during gastric digestion.

1. Introduction

Food processing results in modifications of food properties. These properties include initial chemical and nutritional composition, physical properties and structure, stability of nutrients during storage, as well as release and absorption of beneficial compounds (MacEvilly & Peltola, 2008). Commonly used processing operations for fruits and vegetables include freezing, freeze drying, and convective drying.

Previous studies have shown that freezing modifies fruit initial properties and composition of fruits. For example, freezing of apples (var. *Granny Smith* and *Golden*) and mangos (var. *Kent*) has been shown to modify the fruit texture, color, and physico-chemical (water content, soluble solids content, and pH) parameters (Chassagne-Berces, Fonseca, Citeau, & Marin, 2010; Chassagne-Berces et al., 2009). Mazzeo et al. (2015) observed different color values between frozen asparagus, green beans, and zucchini compared to their fresh counterparts. In contrast, phytochemicals, in particular lutein and flavonoids, were similar in fresh and frozen asparagus, green beans, and zucchini.

In addition to freezing, freeze and convective drying may also influence initial food properties and composition. Freeze drying of apples has been shown to cause a reduction in the reducing sugar content, total sugar content, and total phenol content (Huang, Zhang, Wang, Mujumdar, & Sun, 2012). Both freeze drying (−50 °C, 5 Pa) and convective drying (2 h at 80 °C followed by 6 h at 60 °C) have been shown to increase the antioxidant activity of tomatoes (*Lycopersicon esculentum Mill*) (Chang, Lin, Chang, & Liu, 2006). Convective drying at different temperatures (from 50 °C to 70 °C) has also been shown to cause increases in antioxidant activity in dried orange peel (*Citrus aurantium* v. Canoneta) compared to fresh samples (Garau, Simal, Rosselló, & Femenia, 2007).

The influence of processing on initial food properties might be the result of cellular and structural changes that occur during processing. For example, Delgado and Rubiolo (2005) observed that slow freezing rates (<1.5 °C/min) greatly influenced tissue structure and caused water loss in strawberries (*Fragaria x ananassa*). Additionally, Chassagne-Berces et al. (2009) observed that freezing caused cell membrane breakage, which resulted in cell wall collapse and tissue breakage in *Granny Smith* apples. Freeze drying and convective drying of apples have also been shown to cause cellular changes in the food matrix. Huang et al. (2012) found that freeze drying (−40 °C, 100 Pa) in a microwave vacuum dryer (75–300 W, 5 kPa) resulted in cell wall shrinkage in apples (var. *Red Fuji*). Also, Rodríguez, Santacatalina, et al. (2014) observed cell collapse and cell disruption in apple slices (var. *Granny Smith*) dried with hot air, with more cellular changes occurring at higher (>70 °C) drying temperatures compared to lower drying temperatures (30–60 °C).

In addition to initial composition and quality parameters, processing that results in changes in food nutrient content and cellular

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Nomenclature

AA	antioxidant activity, mg trolox/g dm_o	GAE	gallic acid equivalent
AIR	alcohol insoluble residues, g/100 g dm_o	LM	light microscopy
C	extraction yield, g/g dm_o or g/100 g dm_o	MRE	mean relative error (%)
C_o	initial extraction yield, g/g dm_o or g/100 g dm_o	r^2	coefficient of determination
C_{calc}	calculated value	S_{calc}	standard deviation of the calculated values
C_{eq}	equilibrium extraction yield, g/g dm_o or g/100 g dm_o	S_{exp}	standard deviation of the experimental values
C_{exp}	experimental value	SE	standard error of the estimated parameters
CD	convective drying	SEM	scanning electron microscopy
CI	confidence intervals	SSE	summed square of residuals statistics
dm	dry matter	TPC	total polyphenol content mg GAE/g dm_o
dm_o	initial dry matter	VAR	percentage of explained variance (%)
FD	freeze-drying	α	kinetic reaction constant of the Weibull model s
FN	frozen with liquid nitrogen	β	shape parameter of the Weibull model

structure may also influence the release, bioaccessibility, and bioavailability of nutrients from the food matrix (Parada & Aguilera, 2007). Previous studies have shown that both processing conditions and cellular structure of foods influence the release and absorption of their constituent nutrients. Ellis et al. (2004) showed (*in vivo*) the role of cell walls on the bioavailability of lipids in almond seeds and concluded that intact cell walls prevented the release of intracellular lipids. Furthermore, a theoretical model has been developed relating the bioaccessibility of lipids in almonds to the rupture properties of almond cell walls. This model has been related to the breakdown and size reduction of almond particles during digestion (Grassby et al., 2014).

In fruit and vegetable products, Netzel et al. (2011) found that the liberation of carotenoids, evaluated using an *in vitro* gastric and intestinal digestion model, was higher in a puree of cooked (100 °C, 10 min) or blanched (80 °C, 10 min) carrots compared to fresh carrot puree. Similarly, blanching of carrots (in both water and acidified water (45 g/l citric acid, pH 1.3 at 100 °C for 4 min)) has been shown to promote the release of β-carotene, most likely as a result of cell wall breakdown (Jabbar et al., 2014). Bioaccessibility and bioavailability of β-carotene in carrots has been shown to be influenced by the degree of particle size reduction, heat treatment, and cell wall rupture. Of these factors, cell wall rupture was found to be necessary, prior to release and absorption of β-carotene in carrots. This indicates that cell wall rupture may play a crucial role in nutrient release and absorption in other fruit and vegetable products as well (Tydeman, Parker, Faulks et al., 2010; Tydeman, Parker, Wickham, et al., 2010).Previous studies have shown that processing (freezing, freeze drying and convective drying) may influence both the initial properties and composition as well as the cellular structure of food products. Consequently, modifications in cellular structure of food matrices may result in modification of the release, bioaccessibility, and bioavailability of nutrients from foods. As such, the objective of this study was to evaluate the effects of different processing techniques (freezing, (FN), freeze drying, (FD), and convective drying, (CD)) on the microstructure, chemical characteristics, and release of bioactive compounds from *Granny Smith* apples during *in vitro* gastric digestion.

2. Materials and methods

2.1. Samples

Apples (*Malus domestica* var. *Granny Smith*) were purchased from a local supermarket (initial moisture content of 6.81 ± 0.04 g/g dm and total soluble solids of 12.1 ± 0.5 °Brix). Apples were stored at 4 °C for a maximum of one week. Cubes were cut (0.01 m edge) from the center regions of the apple tissue, not including the peel or core, and immediately processed after cutting.

2.2. Freezing, freeze drying and convective drying processes

Apple cubes were frozen by immersion in liquid nitrogen (FN) (boiling point = −196 °C) until the core temperature reached equilibrium with the freezing temperature (~30 s). Once frozen, samples were thawed in a cold chamber at 4 ± 1 °C for aprox. 45 min prior to *in vitro* digestion.

Freeze drying (FD) was completed using a freeze-drier (Telstar LyoQuest, Barcelona, Spain) operating at −50 °C and (vacuum pressure of 30 Pa) until a final moisture content of 0.05 ± 0.01 kg water/kg dm.

Convective drying (CD) was completed in a laboratory-scale hot air dryer operating at 60 °C with an air velocity of 2 m/s (Garau et al., 2007). Samples were dried until they reached a final moisture content of 0.20 ± 0.03 kg water/kg dm (136.0 ± 0.8 min).

Before *in vitro* digestion, FD and CD samples were rehydrated by immersion in distilled water at 37 °C until they reached a final moisture content similar to raw samples (6.81 ± 0.04 g/g dm). Distilled water was used to rehydrate the apple samples, as this is similar to what may be done prior to consumption of certain dried products.

2.3. In vitro digestion procedure

Apple samples were digested following the *in vitro* gastric digestion method reported by Bornhorst and Singh (2013). Briefly, simulated saliva was prepared by dissolving 1 g/l mucin, 2 g/l α-amylase, 0.117 g/l NaCl, 0.149 g/l KCl, and 2.10 g/l $NaHCO_3$ in deionized water at pH 7.0. Simulated gastric juice was prepared by dissolving 1 g/l pepsin, 1.50 g/l mucin, 8.78 g/l NaCl in deionized water at pH 1.8–2.0. All solutions were prepared daily.

For all processed and raw apples cubes, samples (10–15 g) were mixed with 10 ml of simulated saliva for 30 s, followed by immersion in 100 ml of simulated gastric juice pre-heated to 37 °C. The mixture was incubated in a shaking water bath (Unitronic 320 OR, Barcelona, Spain) at 37 °C and 100 rpm for up to 3 h. Samples were taken initially (no digestion), after mixing with saliva, and after 10, 20, 30, 45, 60, 90, 120, and 180 min of gastric digestion for moisture, acidity, and soluble solid analyses. Samples were taken initially (no digestion), and after 60, 120, and 180 min of gastric digestion for total polyphenol content, and antioxidant activity analyses. Samples were taken initially (no digestion) and after 180 min of gastric digestion for carbohydrate composition and microstructural analyses. All digestion experiments were performed at least in triplicate, and results were expressed in initial