# LWT - Food Science and Technology 65 (2016) 228-236



Contents lists available at ScienceDirect

# LWT - Food Science and Technology

journal homepage: www.elsevier.com/locate/lwt



# Polyphenolic compound stability and antioxidant capacity of apple pomace in an extruded cereal



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# A R T I C L E I N F O

Article history: Received 28 March 2014 Received in revised form 24 July 2015 Accepted 27 July 2015 Available online 30 July 2015

Keywords: Apple pomace Oat Extrusion Antioxidants Cereal Polyphenols

# ABSTRACT

The effect of extrusion temperature and feed moisture content on the phenolic content of apple pomace was evaluated to obtain an extruded cereal high in antioxidants. Mixtures of oat flour, apple pomace and potato starch were conditioned at different moisture contents (23-29 g/100 g) and extruded at different temperatures  $(115-165 \ ^{\circ}\text{C})$  in a single screw extruder. Total phenolic content, individual phenolic compounds, antioxidant activity and lipid oxidation index were determined in extruded products. Compared with the unprocess mixture, a retention between 79.9 and 97.1% of total phenolic content was observed whereas individual phenolic compounds were retained between 57 and 71% for chlorogenic acid, 55-64% for caffeic acid, 38-51% for *p*-coumaric acid, 25-28% for ferulic acid, 56-70% for rutin, and 46-76% for phloridzin. Most of the individual phenolic compounds showed higher stability at  $140 \ ^{\circ}\text{C}$  with intermediate feed moisture content. Antioxidant activity of the extruded products did not show significant changes compared to the unprocessed mixture. Thus, despite the use of high temperatures in the extrusion-cooking it is possible to minimize the loss of bioactive compounds to achieve products, such as ready-to-eat cereals, that contain important antioxidant capacities.

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# 1. Introduction

The development of easy-to-eat products, such as snacks and instant cereals, which are improved by addition of functional components, such as fiber and antioxidants is a growing consumption tendency. These products have been developed through the use of low-cost, versatile technologies, such as extrusioncooking processes (ECPs). ECPs are affected by a combination of several factors, such as moisture content of the starting materials, pressure-temperature, and screw speed, which cause physical and chemical transformations in the final product and therefore affect product quality.

ECP technology has been used in the development of expanded cereals that include the addition of other ingredients, such as vegetables (Bisharat, Oikonomopoulou, Panagiotou, Krokida, & Maroulis, 2013), fruits (Camire, Dougherty, & Briggs, 2007), or pomace derived from fruits (Hwang, Choi, Kim, & Kim, 1998; Khanal, Howard, & Prior, 2009), in order to increase the fiber and antioxidant contents, which may promote health. The use of apples or apple pomace to develop products with high antioxidant contents has been previously reported (Vasantha, Wang, Huber, & Pitts, 2008). Previous studies have shown that approximately 58% of phytochemicals remain in the pomace during industrial apple processing (Guyot, Marnet, Sanoner, & Drilleau, 2003). Phytochemicals contained in apple pulp include catechin, procyanidin,

*Abbreviations:* ECP, Extrusion-cooking process; OF, Oat flour; AP, Apple pomace; PS, Potato starch; UM, Unprocessed mixture; HPLC, High-performance liquid chromatography; DPPH, 2,2-diphenyl-1-picrylhydrazyl; Trolox, 6-hydroxy-2,5,7,8-tetramethylchroman-2-carboxylic acid; TBA, thiobarbituric acid; GAE, Gallic acid equivalents; TE, Trolox equivalents; MDA, malondialdehyde.

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phloridzine, phloretin, glucosides, and ferulic and caffeic acids (Lee, Kim, Kim, Lee, & Lee, 2003; Podsędek, Wilska-Jeszka, Anders, & Markowski, 2000), and it has been reported that the peel also contains quercetin-glucoside and cyanidin-glucoside (Wolfe & Liu, 2003).

In a recent study, it was demonstrated that apple pomace processing may result in changes in antioxidant content and activity (Heras-Ramírez et al., 2012). These effects have been reported using ECP technology, and in some cases, important losses or increases (Khanal et al., 2009; Sharma, Gujral, & Singh, 2012) of phytochemical compounds are attributed to thermal effects and chemical changes due to processing conditions.

Some reports have shown that the thermal degradation of phenolic compounds may be due to complex formation by reaction with Maillard by-products, and high moisture content promotes phenolic compound polymerization (Remy, Fulcrand, Labarbe, Cheynier, & Moutounet, 2000), affecting their extractability and antioxidant activity (Dlamini, Taylor, & Rooney, 2007). In contrast, transformation in more easily extractable forms of phenolic compounds has been reported in single screw extruders with low moisture contents (<15 g/100 g), high shear stress, and high temperatures (Awika, Dykes, Gu, Rooney, & Prior, 2003). All of these chemical changes are associated with structural changes that occur the materials subjected to the extrusion increasing the release of the bioactive compounds contained in the cell wall matrix (Reves, Villarreal, & Cisneros-Zevallos, 2007), making these materials more easily extractable (Zielinski, Michalska, Piskula, & Kozlowska, 2006). Although there are some reports on the use of apple pomace in extrusion processes (Hwang et al., 1998; Karkle, Alavi, & Dogan, 2012), these reports focused on the extruded physical changes, which are related to high shearing force in combination with high temperature and pressure that can efficiently disintegrate the rigid cell walls of apple pomace (Hwang et al., 1998) and could cause degradation or release of chemical compounds. However, the stability or retention of bioactive compounds and antioxidant activity from apple pomace added to extruded products has not been reported. Therefore, the aim of this study was to evaluate the effects of extrusion temperature and feed moisture content on the stability and content of phenolic compounds in extruded products obtained using apple pomace in order to obtain extruded cereals that are higher in antioxidants.

## 2. Materials and methods

# 2.1. Starting materials

Oat flour (OF) and potato starch (PS) were obtained from local markets, and apple pomace (AP) was donated by Food Company in Chihuahua, México. Raw materials were placed in polyethylene bags and stored at room temperature until use. All the starting materials were analyzed for moisture, protein, fat, ash, crude fiber, and ash according to AOAC methods 934.06, 920.52, 920.152, 945.16, 940.26, and 962.09, respectively (AOAC, 1998). Carbohydrates were obtained by difference. Additionally, total phenolic content, antioxidant activity, individual phenolic compounds by HPLC, and lipid oxidation index of AP were determined.

## 2.2. Chemical

(-)Epicatechin, rutin, chlorogenic acid, phloretin 2'-β-D-glucoside (phloridzin), caffeic acid, ferulic acid, Folin-Ciocalteu's phenol reagent,2,2-diphenyl-1-picrylhydrazyl (DPPH<sup>•</sup>), and 6hydroxy-2,5,7,8-tetramethylchroman-2-carboxylic acid (Trolox), were purchased from Sigma–Aldrich (Steinheim, Germany). *p*-Coumaric acid was obtained from Fluka (Buchs, Switerland). High-performance liquid chromatography (HPLC)grade solvents (water, methanol, and acetonitrile) were obtained from J.T. Baker (Mexico City, Mexico). 4,6-Dihydroxy-2mercaptopyrimidine, also known as thiobarbituric acid (TBA) was obtained from Spectrum Chemical (NJ, USA) and 1,1,3,3tetraethoxypropane 97 g/100 mL was purchased from Sigma-Aldrich (Mexico City, Mexico). Other analytical grade solvents used for extractions and reagents were from Sigma-Aldrich (Mexico City, Mexico).

## 2.3. Preparation of mixtures for extrusion

A base mixture (1.5 kg) was formulated with OF (60.2 g/100 g mixture), PS (25.8 g/100 g mixture) and AP (14 g/100 g mixture) the resulting moisture content of the mixture was 8.78 g water/100 g mixture. This base mixture was conditioned at different moisture contents according to the experimental design shown in Table 3. The water added to the mixture was evenly distributed by continuous mixing of the ingredients until the respective moisture content was. The resulting mixtures were packed in sealed plastic bags and refrigerated for 24 h.

## 2.4. Extrusion process

The extrusion experiments were carried out on a model 20 DN single screw laboratory extruder (C.W. Brabender Instruments Inc., NJ, USA). The extruder had a screw diameter of 19 mm; a length to diameter ratio of 20:1; nominal compression ratio of 3:1; and a die opening of 3 mm. The inner barrel had a grooved surface to ensure zero slip at the wall. The barrel was divided into independent electrically heated zones (feed zone and central zone and die) cooled by air. A temperature gradient along the barrel was kept constant at 80, 100 and 120 °C. Screw-operated feed hoppers fed the extruder at 50 rpm. The extrusion parameters comprising the independent variables were temperature at the die end of the barrel (104, 115, 140, 165, and 175 °C) and feed moisture content (21, 23, 26, 29, and 30 g/100 g, wet basis).

Before extrusion, experimental mixtures were brought to approximately room temperature (25 °C) and mixed to ensure even moisture distribution. The order of processing was chosen by randomizing feed moisture levels and die end temperatures at a constant screw speed (180 rpm). Each extrusion run was brought to steady state as indicated by constant torque and melt temperatures before sampling and data collection. The extrudates of each mixture were dried at 70 °C for 60 min in a cabinet dryer with an air cross-flow velocity until a final moisture content of approximately 0.136–0.583 kgH<sub>2</sub>O/kg dry matter (dm) was obtained, and then the extrudates were stored at room temperature (25 °C) until evaluation.

## 2.5. Chemical characterization of extruded products

### 2.5.1. Polyphenol extraction

Five g of each powdered extruded sample and raw materials was suspended with 80 g/100 mL acetone solution and sonicated using

Table 1	
Process variables and levels used in the experimental design.	

Input process variables	Levels				
	-1.4142	-1.0	0	+1.0	+1.4142
Extruder temperature (°C)	104.64	115	140	165	175.36
Feed moisture content (g/100 g)	21.76	23	26	29	30.24

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