



## Investigation on the extractability of melanoidins in portioned espresso coffee



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### ABSTRACT

Coffee melanoidins have attracted interest as a result of its potential health benefits. This investigation aims to elucidate the extraction behavior of melanoidins and their populations during the preparation of portioned espresso coffee and its relationship with the antioxidant activity of the coffee brew. Filter-paper pods, FAP capsule, and clone capsule containing light roasted coffee have been investigated. An accumulative fractionation approach has applied to model the extraction kinetics of melanoidins, melanoidin populations, browning, chlorogenic acids (CGA), and antioxidant activity. Melanoidins were very efficiently extracted in clone capsules since less than 9 s was necessary to extract the 50% of the melanoidin content as compared with pods and FAP capsules, and the kinetic of extraction is slower than CGA. The extraction profile of melanoidins and browning fitted better with the antioxidant capacity than CGA and total solids profile. Melanoidin populations were obtained according to ethanol solubility. Total melanoidin content and the ratio between melanoidin populations did not change during extraction volume for espresso coffee. Melanoidin populations soluble at 75% ethanol showed the highest antioxidant activity. However, melanoidins with higher antioxidant activity are extracted at higher volumes. This investigation could make possible the adjustment of the technological requirements of espresso coffeemakers to produce an espresso coffee with high levels of beneficial compounds.

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## 1. Introduction

In addition to the stimulatory properties, coffee consumption is related to certain health benefits, such as anti-inflammatory and antimutagenic effects, reduction of cardiovascular risk, rheumatologic diseases, endometrial tumors, Parkinson's disease, Alzheimer's disease, and the regulation of insulin or body weight, among other (Bichler et al., 2007; Cavin et al., 2008; Lindsay et al., 2002; Morales, Somoza, & Fogliano, 2012; Ranheim & Halvorsen, 2005; Salazar-Martinez et al., 2004; Wang, Qian, & Yao, 2011). Coffee brew is a source of caffeine, hydroxycinnamates, particularly chlorogenic acids (CGA), antioxidants and indigestible fiber (Díaz-Rubio & Saura-Calixto, 2007; Farah & Donangelo, 2006; Manach, Scalbert, Morand, Rémésy, & Jiménez, 2004). Some of the positive physiological effects of coffee consumption have been explained by the antioxidant properties of its constituents, such as the presence of phenolic compounds and Maillard reaction products (Borrelli, Visconti, Mennella, Anese, & Fogliano, 2002; Daglia, Papetti, Gregotti, Bertè, & Gazzani, 2000; Delgado-Andrade, Rufián-Henares, &

Morales, 2005). Human trials considering the Italian consumer average coffee intake (5 cups of espresso per day) found that coffee was able to significantly increase plasma glutathione concentration (Esposito et al., 2003). The contribution of coffee consumption to the overall intake of antioxidants may reach up to 70% in Western diets (Torres & Farah, 2010) but also represents a significant portion of daily intake of fiber (Díaz-Rubio & Saura-Calixto, 2007).

During the roasting of coffee green beans chemical and structural changes take place as a result of the Maillard reaction, caramelization and pyrolysis. The first consequence of roasting is the loss of thermolabile compounds such as trigonelline and chlorogenic acids in a different extent according to the roasting degree and also the formation of new compounds such as melanoidins (Farah, de Paulis, Trugo, & Martin, 2005; Illy & Viani, 2005). Melanoidins are high molecular weight (HMW) Maillard reaction end-products, which are a heterogeneous mixture of brown colored, nitrogen containing polymers, formed through the reaction of reducing sugars with proteins/amino acids (Moreira, Nunes, Domingues, & Coimbra, 2012). Coffee polysaccharides, galactomannan-like and arabinogalactan-like carbohydrates, proteins, and phenolic compounds, mainly hydroxycinnamates, contribute to the formation of coffee melanoidins (Bekedam, Schols, van Boekel, & Smit, 2008; Moreira et al., 2012; Nunes & Coimbra, 2007). Coffee brew is one of the main sources of melanoidins in the human diet and the

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intake of coffee melanoidins estimated to range between 0.5 and 2.0 g per day for moderate and heavy consumers, respectively (Fogliano & Morales, 2011). The impact of melanoidins to coffee brew is not limited to their color contribution since these molecules are involved in the modulation of flavor release, antioxidant and metal-chelating properties, and dietary fiber behavior (Hofmann, Czerny, Calligaris, & Schieberle, 2001; Morales et al., 2012; Moreira et al., 2012; Reichardt, Gniechwitz, Steinhart, Bunzel, & Blaut, 2009). Therefore, melanoidins (including also melanoproteins from bakery products) may contribute 20.2% to the daily antioxidant capacity intake, more than 50% corresponding directly to coffee melanoidins (Pastoriza & Rufián-Henares, 2014). Silván, Morales, and Saura-Calixto (2010) suggested a new definition of coffee melanoidins as a maillardized fiber with potential beneficial effects on gastrointestinal health by combining the antioxidant activity of melanoidins and the low transit of these polymers (Borrelli, Esposito, Napolitano, Ritieni, & Fogliano, 2004). Indeed, condensation of phenolic compounds with indigestible polysaccharides and melanoidins present in coffee could explain the association between coffee consumption and the antioxidant capacity of feces from healthy subjects (Garsetti, Pellegrini, Baggio, & Brighenti, 2000).

There are many different devices and methods to produce coffee brew, which are generally characterized by extraction pressure, extraction process, extract/cup volume, and solid content (Parenti et al., 2014; Petracco, 2005). There is not established an official definition for espresso coffee but it could be described as the brew obtained by percolation of hot water under pressure through compacted cake of roasted ground coffee, where the energy of the water pressure is spent within the cake (Illy & Viani, 2005). During espresso brewing, water at approximately 9 bars and 88–92 °C is forced to go through coffee grounds compacted in a small brewing chamber (coffee cake) (Albanese, Di Matteo, Poiana, & Spagnamusso, 2009). Temperature, pressure, brewing time, coarse grind and ground coffee portion, cake shape and moistening, and water quality, among others, have an influence on the physical and sensorial properties of the espresso coffee and the effectiveness of the extraction process (Albanese et al., 2009; Petracco, 2005). The overall quality of espresso coffee depends largely on the quality of the roasted coffee, but also on the extraction procedure.

Extensive studies have been focused on the effect of different physical variables during the percolation process on the extraction of caffeine, CGA, key aroma compounds and sensory analysis (Andueza, de Peña, & Cid, 2003; Andueza et al., 2002; Gloess et al., 2013; Illy & Viani, 2005; López-Galilea, de Peña, & Cid, 2007; López-Galilea, Fournier, Cid, & Guichard, 2006; Maeztu et al., 2001). Ludwig et al. (2012) investigated the extraction behavior of the main coffee antioxidants (caffeoylquinic acids, melanoidins, and caffeine) and the antioxidant capacity, during brewing time in the most widely consumed coffee brew methods (filter and espresso) in coffee. This investigation concluded that more than 70% of the antioxidants (except dicaffeoylquinic acid) of an espresso coffee were extracted during the first 8 s.

Nowadays, portioned coffee is being well accepted by consumer and a number of commercial brands of single-dose and formats are already present in the market. Recently, Mestdagh, Davidek, Chaumonteuil, Folmer, and Blank (2014) reported the kinetics of coffee aroma extraction during espresso coffee preparation by applying an accumulative approach for fraction collection. To our knowledge, investigation on the extraction of melanoidins in filtered and espresso coffee is scarce, and they were considered as the browning rate and samples where sequentially collected during coffee brewing (Ludwig et al., 2012). Then a complete understanding of the kinetic of melanoidins extraction during preparation of espresso coffee is missing. Since melanoidins have attracted huge interest as a result of claims for potential health benefits, antioxidant activity and sources of dietary fiber, this investigation aims to elucidate the extraction behavior of melanoidins and their populations during the preparation of portioned espresso coffee. In addition, the antioxidant capacity related to the melanoidins extraction, browning and CGA content is also investigated.

## 2. Materials and methods

### 2.1. Materials

Four portioned coffee samples were supplied by three Spanish coffee companies. Single-dose samples were freshly produced, commercially available and identified as gourmet coffees roasted under light-medium conditions. Sample F was filter-paper pods hermetically sealed between a two fine layers of filter paper and individually packaged in a protected atmosphere. Sample B was FAP capsule packaged into a biodegradable plastic capsule in a protected atmosphere, which preserves the quality of the ground coffee by protecting it against moisture and oxidation processes. Samples NC and NB were clone capsules closed with aluminum foil (Nespresso® compatible). Pure Arabica coffee (*Coffea arabica*) from Colombia was used for samples F, B, and NC. An Arabica and Robusta coffee (*Coffea canephora*) blend from Vietnam, Guatemala, and Brazil at percentage 35:65 was used for sample NB. Samples were stored at 12–16 °C and kept 1 h at ambient temperature before use. According to the coffee companies, the grinding grade of the samples was the most adequate to each type of container and coffee machine. Technical characteristics of the espresso coffeemakers, containers, and coffee are summarized in Table 1.

### 2.2. Preparation of coffee brew

Extraction was performed with three commercial espresso coffee machines and customized to filter-paper pods (F), FAP capsules (B), and clone capsules (N) as described in Table 1. Mineral water (Solana de Cabras, Cuenca, Spain) was used with next characteristics (mg/L): 260 dry matter, 284 bicarbonate, 5.1 sodium, 1.1 potassium, 7.4 chloride, 21.3 sulfate, 59.4 calcium, and 7.2 silica. Coffee brew fractions F1, F2, F3, and F4 were collected with final volumes of 5, 9, 16, and 46 mL for coffee samples F, B, NC, and NB. Samples were collected in marked containers for volume. In addition, a fifth fraction of 115 mL was collected for samples F and B, and 85 mL for coffee samples NB and NC. Coffee machine N has a preselected volume of 46 mL for espresso coffee and 96 mL for Longo coffee. Coffeemakers F and B had a variable control of the volume. Accumulative fraction collection (each fraction contains the previous ones plus certain additional volume) was chosen for higher precision than the sequential collection. Fraction F4 and F5 are also identified as conventional espresso coffee and Longo coffee, respectively. Percolation times ranged from 3 to 80 s regarding the espresso machine and fraction collected. The manufacturer instructions for brewing and adequate machine settings, including preheating, were followed before each coffee preparation. At least an average of 10 extractions were performed and pooled for each fraction. Coffee fractions were lyophilized (VirTis Benchtop-6 KB, SP-Scientific, Ipswich, UK) and stored at –18 °C until use. In other case, coffee brew was freeze from three different extractions. The temperature profiles ( $n = 3$ ) were registered by digital thermometer (Model HD2107.1, Delta OHM SRL, Italy) linked to two thermocouples K type ( $\varnothing = 0.5$  mm) located at the exit and at bottom of the cup.

### 2.3. Basic determinations

pH was determined using an electronic pH meter (Schott model CG-837, Mainz, Germany). Total solids content in coffee brew and fractions were determined gravimetrically after sample lyophilization of coffee brew. Additionally, total solid content in fraction F4 and F5 was calculated by oven drying 25 mL of coffee brew at 105 °C until constant weight (18 h). The rate of extraction was defined as the percentage of total solids with respect to ground roasted coffee dosage in the container. The maximum rate of extraction in pods and capsules was calculated after manually withdrawing of ground coffee in the container ( $n = 5$ ) and manually extracting the ground coffee (7 g) with 100 mL of water at 80 °C under stirring for 5 min. The resulting coffee brew was filtered

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