Journal of Food Engineering 120 (2014) 158–166

Contents lists available at ScienceDirect

Journal of Food Engineering

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

Block freeze-concentration of coffee extract: Effect of freezing and thawing stages on solute recovery and bioactive compounds

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ARTICLE INFO

Article history:
Received 28 May 2013
Received in revised form 17 July 2013
Accepted 21 July 2013
Available online 6 August 2013

Keywords:
Cryoconcentration
Solute yield
Coffee
Chlorogenic acids
Antioxidant activity

ABSTRACT

Coffee extract was freeze-concentrated using the total block technique. The effects of four parameters were evaluated: the initial coffee mass fraction (5 and 15% w/w), the cooling temperature (−10 and −20 °C), the heating temperature (20 and 40 °C) and the freezing direction (parallel and counter-flow to the thawing direction). The solid concentration was measured during the thawing stage to quantify the solute recovery and the concentration index for one stage of freeze concentration. The coffee mass fraction, the freezing direction and the cooling temperature significantly influenced the solute recovery. A concentration index between 1 and 2.3 was obtained in one cycle. The effect of block freeze concentration on the bioactive compound concentration and the antioxidant activity was measured. The coffee bioactive compounds were distributed in proportion to the total solid content in the ice and liquid. Therefore, block freeze concentration is an effective technique to preserve functional properties of coffee extracts.

1. Introduction

Coffee is the most traded food in the world, and its production has great economic and social importance worldwide (Esquivel and Jiménez, 2012; Vignoli et al., 2011). For the consumer, the value of coffee is provided by its sensory and functional properties; for this reason, technologies that promote quality preservation are highly valued in coffee processing. In the production of freeze-dried coffee, freeze concentration (FC) technology is used to remove water from coffee extracts to increase the solid content and reduce the time and cost of the freeze-drying process. At the same time, the sensory properties of the product are preserved using low temperatures (Boss et al., 2004; Joët et al., 2010; Sánchez et al., 2009).

Water removal in FC is achieved by cooling the solution until the ice crystals form and separate (Miyawaki et al., 2005). Three techniques are used according to the ice crystal growth: suspension FC, film FC (progressive or falling film FC) and block FC (total or partial) (Aider and de Halleux, 2009; Sánchez et al., 2009). Suspension FC is a unique technique implemented at the industrial level. Different techniques, such as falling film FC (Chen et al., 1998; Sánchez et al., 2011), progressive FC (Miyawaki et al., 2005) and block FC (Aider and Ounis, 2012; Nakagawa et al., 2010a), are being developed to reduce operational costs.

In the block FC method, also known as freeze–thaw concentration, the solution to be concentrated is completely frozen and then partially thawed to recover a fraction of liquid with a higher concentration (Aider and de Halleux, 2009; Nakagawa et al., 2010b). Block FC consists of three stages: freezing, thawing and separation of the concentrated liquid fraction (Moreno et al., 2013). These stages define the separation efficiency (Nakagawa et al., 2009). Additionally, the process can be repeated in successive cycles to increase the concentration index (Aider and Ounis, 2012).

The technical viability of the block FC method has been proposed recently by several researchers (Gao et al., 2009; Nakagawa et al., 2010a; Aider and Ounis, 2012; Boaventura et al., 2012; Miyawaki et al., 2012; Petzold et al., 2013). During the freezing stage, heat and mass transfer phenomena can modify the solute occlusion, which should be as low as possible. Chen et al. (2001) eported that the solute elution in the freezing front in FC depends on the molecular size of the compounds. Certain authors have reported that the solute separation is controlled by the thawing stage (Nakagawa et al., 2010b). For coffee solutions, Moreno et al. (2013) studied the use of aids in the separation stage. These authors reported the influence of the FC protocol and solution type on solute recovery and the concentration index; for this reason, there is no agreement on the significance of the process variables. The effects of the process variables of block FC on the separation efficiency of coffee extracts have not been reported.

Coffee can be considered to be a functional beverage due to its radical scavenging capabilities (Cheong et al., 2013; Esquivel and

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http://dx.doi.org/10.1016/j.jfoodeng.2013.07.034

Nomenclature

CI	concentration index
CI_{cum}	cumulative concentration index
C_{FCL}	concentration of bioactive compounds in the freeze-concentrated liquid
C_{RI}	concentration of bioactive compounds in the residual ice
f	thawing fraction
F_D	freezing direction
IL	ice loss percentage
m_{s0}	initial solute mass
$m_{s\,liq}$	solute mass in the liquid fraction
m_0	initial mass
m_{liq}	collected liquid mass
T_C	cooling temperature
T_H	heating temperature
X_{s0}	coffee mass fraction in the initial solution
X_S	coffee mass fraction
$X_{s\,liq}$	coffee mass fraction in the freeze-concentrated liquid fraction
Y	solute yield

Jiménez, 2012). Several studies have reported the health benefits of coffee consumption related to the components with antioxidant activity, such as the group of chlorogenic acids and caffeine. Chlorogenic acid (3-caffeoylquinic acid), cryptochlorogenic acid (4-caffeoylquinic acid), neoclorogenic acid (5-caffeoylquinic acid) and caffeine are the major bioactive compounds present in coffee (Ferruzzi, 2010; Fujioka and Shibamoto, 2008; Sopelana et al., 2013; Vignoli et al., 2011). The block FC method has been shown to retain nutritional and functional properties of the product using low processing temperatures (Belén et al., 2013; Boaventura et al., 2012); however, this effect has not been tested for coffee extracts.

The aim of the present study was to evaluate the effect of the initial coffee mass fraction, the cooling temperature, the heating temperature and the freezing direction on the solute yield and concentration index of block freeze-concentrated coffee extracts. Additionally, the impact of the technique on bioactive compound concentration and the antioxidant activity of the coffee extract was tested.

2. Materials and methods

2.1. Materials

Coffee solutions were prepared from freeze-dried soluble coffee supplied by the company Buencafé Liofilizado de Colombia (Colombian Coffee Growers Federation, Colombia) for the FC tests. The coffee was added to distilled water at 35 °C and mixed for 20 min. The samples were stored at 4 °C for 12 h. The solid concentration is expressed as the coffee mass fraction (X_S), which is defined as the mass of coffee solids per unit of coffee solution mass. The relationship between Brix degrees and X_S is represented by the equation $X_S = 0.0087$ °Brix ($R^2 = 0.991$). This expression was obtained by preparing coffee solutions at 10, 20, 30, 40 and 50 °Brix and by measuring coffee mass fraction using the weight loss technique in the oven at 103 °C for 4 h according to technical standard NTC4602 (Icontec, 2009). The measurements were performed in triplicate. The coffee mass fraction of the solutions was ascertained immediately before the FC tests by refractometry (Atago Pal 100, Japan). A liquid coffee extract was used for the measurement of bioactive compounds. This extract belonged to the same batch of soluble coffee and was also provided by Buencafé Liofilizado de Colombia.

2.2. Methods

2.2.1. Freeze concentration protocol

The effects of the initial coffee mass fraction (X_S), cooling temperature (T_C), heating temperature (T_H) and the freezing direction (F_D) were studied. A full factorial design with four factors and two levels was used for a total number of 16 tests (Table 1). The coffee solutions were subjected to one cycle of freezing, thawing and separation to study the effect of process variables on solute yield after one cycle of FC.

The block FC device is shown in Fig. 1. In total, 160 g of the coffee sample was placed into a cylindrical container (1) measuring 52.5 mm in diameter and 85 mm in height. The container is a double jacket device for the flux of cooling and heating fluids. The internal jacket is 19 mm in diameter (2). The cooling/heating fluid was a mixture of ethylene glycol and water (53% w/w) coming from two circulated baths (4 and 5) (Polystat, Cole Parmer, USA). The baths were temperature controlled (6 and 7) at an interval from −35 °C to 150 °C ± 0.01 °C. The baths pumped the heat exchange fluid to the jackets through a system of ducts and valves (7).

During the tests, the heat exchange fluid temperature was settled in one bath. After the fluid reached the temperature, it was circulated to the jackets to freeze the solution inside. The heat transfer was in the radial direction from the internal wall (for freezing parallel to thawing) or from the external wall (for freezing in counter-flow to thawing). Meanwhile, the heating temperature of the second bath was settled. When the sample was frozen and the temperature was approximately constant, the thawing stage was begun by pumping the heating fluid through the external jacket. The exit valve (9) was opened and the liquid fraction was separated in a collector vessel (10) on a scale (11) (Ohaus PA3102, USA) with a capacity of 3100 g and a precision of 0.01 g for weight measurement. During the thawing stage, the temperature of the internal jacket was maintained one Celsius degree below the

Table 1
Experimental design.

Test	X_S	T_C	T_H	F_D
1	0.05	−10	20	1
2	0.05	−10	20	−1
3	0.05	−10	40	1
4	0.05	−10	40	−1
5	0.05	−20	20	1
6	0.05	−20	20	−1
7	0.05	−20	40	1
8	0.05	−20	40	−1
9	0.15	−10	20	1
10	0.15	−10	20	−1
11	0.15	−10	40	1
12	0.15	−10	40	−1
13	0.15	−20	20	1
14	0.15	−20	20	−1
15	0.15	−20	40	1
16	0.15	−20	40	−1

F_D + 1: counter-flow to thawing; F_D-1: parallel to thawing.