Journal of Food Engineering 166 (2015) 64–71

Contents lists available at ScienceDirect

Journal of Food Engineering

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

Volatile compounds, sensory quality and ice morphology in falling-film and block freeze concentration of coffee extract

F.L. Moreno [a,b,d], M.X. Quintanilla-Carvajal [b], L.I. Sotelo [b], C. Osorio [c], M. Raventós [d], E. Hernández [b], Y. Ruiz [b,*]

[a] Biosciences Doctoral Program, Universidad de La Sabana, Campus Universitario del Puente del Común, km 7 Autopista Norte de Bogotá, Chía, Cundinamarca, Colombia
[b] Agro-industrial Process Engineering, Universidad de La Sabana, Campus Universitario del Puente del Común, km 7 Autopista Norte de Bogotá, Chía, Cundinamarca, Colombia
[c] Departamento de Química, Universidad Nacional de Colombia, AA 14490, Bogotá, Colombia
[d] Agri-Food Engineering and Biotechnology Department, Universidad Politécnica de Cataluña (UPC), C/Esteve Terradas, 8, 08860 Castelldefels, Barcelona, Spain

ARTICLE INFO

Article history:
Received 27 January 2015
Received in revised form 28 April 2015
Accepted 12 May 2015
Available online 13 May 2015

Keywords:
Coffee concentration
Volatiles
Cryoconcentration
Sensory evaluation
Ice morphology

ABSTRACT

Coffee extract was freeze-concentrated through block and falling-film techniques. Solute retention and concentration efficiency were determined after one stage of these processes. Ice morphology was characterized through image analysis. Preservation of volatile compounds was determined through GC–MS. The effect of coffee extract on flavour was determined after freeze concentration through sensory evaluation. Solute occlusion was higher for falling-film than for block freeze-concentration, with an average distribution coefficient of 0.45 and 0.29, respectively. The ice crystal size was lower for the falling-film technique; this explains the higher solute occlusion. The dewatering capacity was higher for the falling-film technique, as this process is faster than block freeze-concentration. The most abundant volatile compounds of the coffee extracts were preserved after freeze concentration with both techniques. In the same way, no differences were found in most of the sensory attributes of the freeze-concentrated extract obtained using both techniques. Our results confirm the benefits of the block and falling-film freeze concentration techniques in preserving the quality of coffee extracts.

1. Introduction

Coffee is the most traded food in the world, and the preservation of its flavour is very important during processing (Caporaso et al., 2014). Freeze-dried coffee is a higher quality product than soluble coffee due to its lower processing temperatures (Fissore et al., 2014). The process of obtaining freeze-dried coffee starts with an initial stage of aqueous extraction by percolation, followed by the concentration and drying of the extract. The objective of the concentration stage is to eliminate some of the water from the extract. This stage is performed through freeze concentration. Freeze concentration (FC) is a technology used to remove water from aqueous solutions by freezing (Sánchez et al., 2009) to reduce processing times (Moreno et al., 2014c).

Three techniques of freeze concentration are recognized: suspension FC, film FC (known as progressive FC) and block freeze concentration (known as freeze–thaw FC) (Moreno et al., 2014a). The most implemented FC technique is the suspension FC. In this technique, small ice crystals are produced in a scraped heat exchanger and then removed through washing columns (Qin et al., 2009). A high separation efficiency is achieved; however, the initial and operational costs are relatively high (Petzold and Aguilera, 2013). As a result, different techniques, such as falling-film and block FC, are being developed. In block freeze-concentration the solution is completely frozen in a vessel. Then, the solution is partially thawed to recover a concentrated liquid fraction (Aider and de Halleux, 2009; Moreno et al., 2014c; Nakagawa et al., 2010a,b). On the other hand, in falling-film freeze concentration (FFFC), the solution is circulated through a cooling plate. An ice sheet is formed on the plate and the solution is recirculated until a desired concentration is reached in a batch operation (Hernández et al., 2010). Several researchers have established the viability of both techniques to concentrate food solutions (Aider and de Halleux, 2008; Nakagawa et al., 2010a,b; Petzold and Aguilera, 2013; Raventós et al., 2007; Sánchez et al., 2010; Petzold et al., 2015; Benedetti et al., 2015).

The behaviour of block and falling film freeze concentration is related to some parameters such as the solute occlusion in the ice, the effect of the technique on volatile compounds and the effect on sensory quality of the product. The separation efficiency in freeze concentration (FC) is related to solid occlusion in the ice

---

* Corresponding author. Tel.: +57 1 8615555x25217.
E-mail address: ruth.ruiz@unisabana.edu.co (Y. Ruiz).

http://dx.doi.org/10.1016/j.jfoodeng.2015.05.018
0260-8774/

layer that can be influenced by the morphology of ice crystals created by the freezing conditions and the solution type (Ayel et al., 2006; Butler, 2002; Okawa et al., 2009; Pardo et al., 2002). Understanding the quantitative relationships between freeze concentration and ice crystal morphology is of practical importance. The physical properties of the ice layer can be considered to be the result of the characteristics (size) and spatial arrangement of the crystals (Germain and Aguilera, 2012). The freezing stage is a key step because it fixes the morphology of the frozen material, and as a result, it can affect the efficiency of the freeze concentration process, which is why it is important to evaluate the morphology generated by the processes mentioned above. It has been observed that the distribution of ice crystals of different sizes depends not only on the freezing rates but also on the sample size and freezing direction, among other variables (Hottot et al., 2007). On the other hand, the sensory attributes of the coffee beverage is one of its most important quality parameters, consequently, sensory analysis is the most used technique to evaluate coffee quality (Cheong et al., 2013; MacLeod et al., 2006; Sopelana et al., 2013; Farah et al., 2006). However, there are few reported works on the efficacy of volatile compound preservation when using FC (Ramos et al., 2005).

Block and falling-film FC have been studied in coffee extract concentration (Moreno et al., 2014a). However, the effect of FC on volatile compounds and the sensory quality of the beverage has not been studied in these techniques. In addition, the ice morphology achieved through these FC techniques and its relationship with solute occlusion has not been determined. Thus, the aim of the present study was to compare the solute retention, dewatering capacity per unit of time of the operation, ice morphology, volatile compound preservation, and sensory quality between falling-film freeze concentration and block freeze-concentration of a coffee extract.

2. Materials and methods

2.1. Materials

The coffee extract from roasted Colombian coffea arabica supplied by the company Buencafé Liofilizado de Colombia (Colombian Coffee Growers Federation) produced at industrial conditions was used for the FC tests. The extract was 13% w/w solids. The extract was stored at −18 °C and thawed at 4 °C for 8 h prior to the tests.

2.2. Freeze concentration tests

2.2.1. Block freeze-concentration tests

One stage of block FC was tested according to the parameters reported by Moreno et al. (2014c). The block FC technique consisted of the complete freezing of the extract in a closed vessel and the subsequent partial thawing and separation of the liquid phase. The tests were performed in the device shown in Fig. 1a. The coffee sample (160 g) was placed into a cylindrical double-jacketed container that was 5.2 cm in diameter and 8.5 cm in height. The internal jacket was cooled through a mixture of ethylene glycol and water (53% w/w) from a bath (Polystat, Cole Parmer, USA) with temperature control (−35 °C to 150 °C ± 0.01 °C). The cooling fluid temperature was set at −10 °C. The cooling fluid was circulated into the internal jacket once it reached that temperature.

Ice grew from the centre to the external wall of the container. When the sample was completely frozen, it was thawed by heating the external jacket at 20 °C with the fluid from the second bath. Fifty percent of the extract mass was collected and separated from the ice, according to the results proposed by Moreno et al. (2014c). The solid concentration (Cs) of the liquid and the ice fractions was measured via refractometry (Atago Pal 100, Japan). Cs is defined as the mass of coffee solids per unit of mass of coffee solution. The relationship between Brix degrees and CS is represented by the equation Cs = 0.87 °Brix, as reported by Moreno et al. (2015). The two fractions were stored at 4 °C for 12 h for the analysis of volatile compounds. The tests were performed in triplicate.

2.2.2. Falling film freeze concentration tests

One stage of FFFC was tested according to the parameters reported by Moreno et al., 2014b. The FFFC remained in continuous circulation of the extract through a refrigerated plate until ice sheet formation and separation occurred. The coffee extract (800 g) was freeze-concentrated using the experimental setup outlined in Fig. 1b. The coffee extract flowed through a descending film over a cooling plate that was 25 cm in width and 20 cm in height. The extract was collected in a tank and recirculated by a VGC-400 peristaltic pump (Seditesa, Spain). The cooling fluid temperature was set at −20 °C. The cooling fluid was circulated into the plate once it reached that temperature. Ice grew on the surface of the freezing plate and was removed at the end of the stage. This stage was completed after 2 h when the ice produced was close to 50% of the initial extract. The solid concentration of the liquid fraction and the ice were measured through refractometry (Atago Pal 100, Japan). The temperature of the liquid phase was determined during the tests. The experiments were performed in triplicate.

2.2.3. Data analysis for FC tests

Solute retention in the ice produced through block and falling-film FC was analysed by comparing the solute yield, the concentration index, the partition coefficient, the ice front growth and the dewatering capacity of the operation.

Solute yield (Y) represents the amount of solute or coffee solids recovered in the liquid fraction. It was defined as the relationship

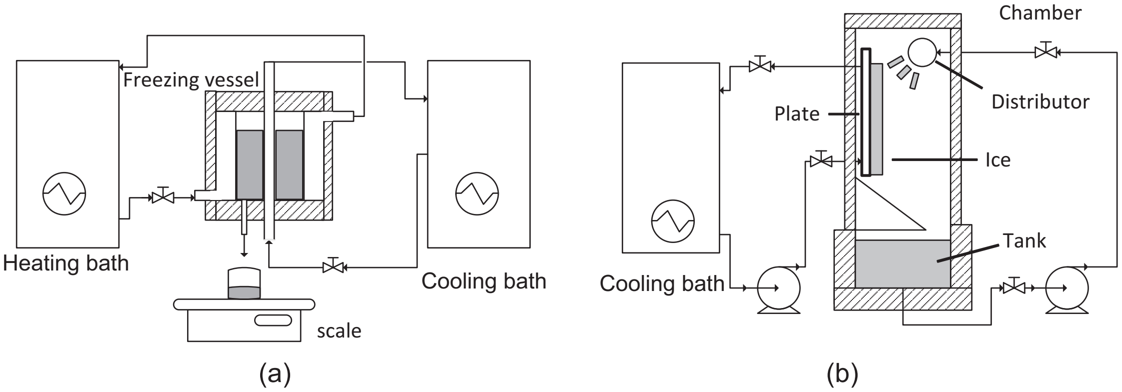

Fig. 1. Experimental setup. (a) Block freeze concentrator; (b) falling-film freeze concentrator.