



A process to concentrate coffee extract by the integration of falling film and block freeze-concentration



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ABSTRACT

A process to concentrate aqueous coffee extract by freeze concentration is proposed to achieve an industrially viable system. The techniques of falling film freeze concentration, fractionated thawing and block freeze concentration were studied. Batches of 40 kg of coffee extract with 5% initial solid concentration were freeze-concentrated in seven stages in a falling film multi-plate freeze concentrator. The ice from each stage was fractionally thawed to recover the coffee solids retained in the ice. The diluted fractions of the thawing stage were freeze-concentrated using the block technique. A concentrated extract with 32.6% solids and an effluent with 0.27% solids were obtained through the integration of these techniques. A concentration index of 6.5, a concentration efficiency of 99.2% and a solute yield of 95% were obtained. The integration of these simple techniques results in a concentration index and solute yield comparable to industrial standards in freeze-concentrated coffee extract production.

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1. Introduction

Coffee is one of the most consumed beverage worldwide (Esquivel and Jiménez, 2012; Sopelana et al., 2011). Freeze-dried coffee is a high-quality product of the coffee industry because of the flavour preservation due to the low-temperature processing conditions (MacLeod et al., 2006). The process of obtaining freeze-dried coffee begins with the extraction of roasted coffee beans by percolation. Subsequently, the extract is freeze-concentrated to remove part of the water and to obtain a concentrated extract. The concentrated extract may be the final product or may be freeze-dried to remove the remaining water to obtain soluble coffee (Boss et al., 2004). The use of freeze-concentration technology is justified by the reduction of the freeze-drying process costs by 25% (Van Pelt and Bassoli, 1990). In addition, the quality of the product is preserved by low processing temperatures (Rahman et al., 2007).

Freeze concentration (FC) is a technique used to remove water from food fluids by freezing (Sánchez et al., 2009). The solution is cooled below the freezing point to produce and separate ice crystals. Three techniques are used for growth of ice crystals, suspension FC, film FC (progressive or falling film FC) and block FC (also known as freeze-thaw FC) (Aider and de Halleux, 2009; Sánchez et al., 2011a).

The only technique implemented in coffee processing at the industrial level is suspension FC. This is an efficient technique in

terms of ice purity and increased concentration (Qin et al., 2007, 2006; van der Ham et al., 2004). With this technique, it is possible to concentrate the coffee extract to 32–35% solids and to obtain a high-purity effluent with 0.1% solids (Van Mil and Bouman, 1990; Van Pelt and Bassoli, 1990). However, this technique requires complicated systems of ice separation and many moving parts, which increases the initial and operating costs (Aider and de Halleux, 2009; Miyawaki et al., 2005; Sánchez et al., 2009). For this reason, several other FC techniques have been studied. Recently, the industrial future of freeze concentration has shifted toward the configuration of one-step systems or a combination of systems rather than suspension freeze concentration because of the simpler separation step (Petzold and Aguilera, 2009, 2013).

In falling film freeze concentration (FFFC), the solution to be concentrated is in contact with a cooled vertical plate on which the fluid descends. The ice forms a single layer on the cold surface, and the solution is re-circulated continuously (Sánchez et al., 2011b). FFFC has been studied with several food fluids (Belén et al., 2013; Chen et al., 1998; Hernández et al., 2009, 2010; Raventós et al., 2007; Sánchez et al., 2010). Flesland (1995) proposed multi-stage FFFC coupled to reverse osmosis for water desalination. In that study, water elimination was efficient. Recently, the recovery of solutes of sucrose solutions retained in ice was attempted using fractionated thawing of the ice (Gulfo et al., 2013; Miyawaki et al., 2012).

In contrast, in block freeze concentration (block FC), the whole solution is frozen and partially thawed to recover the concentrated liquid fraction (Aider and de Halleux, 2009). Block FC has been

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used for sucrose solutions, dairy products, syrup, mate extract and fruit juices (Aider and Ounis, 2012; Aider et al., 2009; Boaventura et al., 2012; Nakagawa et al., 2010a,b). For coffee extracts, Moreno et al. (2014) and Moreno et al. (2013) studied the effect of process conditions on the freezing and thawing stages of block FC. The viability of the technique was primarily demonstrated for low solid concentrations.

In some food applications, FC can be used to maximise the final solid content of the solution. However, in the coffee industry, the minimisation of the solid content of the final effluent is also important due to the high value of the product. Currently, there is no plan to use FFFC or block FC to obtain an extract with a high solid concentration and an effluent with a low solid content that comply with industrial requirements.

The aim of the present study was to propose a process to freeze-concentrate coffee extracts through the integration of falling film freeze concentration, which includes coffee solids recovery by fractionated thawing and block freeze concentration techniques.

2. Materials and methods

2.1. Materials

A coffee extract with 5% (w/w) wet basis of total solid content was prepared from freeze-dried soluble coffee supplied by Buencafé Liofilizado de Colombia (Colombian Coffee Growers Federation, Colombia) and water at 35 °C. The solution was stored at 4 °C for 24 h prior to the tests.

2.2. Methods

Three techniques were studied for coffee extract freeze-concentration following the flowchart shown in Fig. 1. First, the initial extract was freeze-concentrated by the falling film technique. Second, the ice formed in the first technique was thawed fractionally to study the recovery of the retained solutes. Finally, the diluted fractions obtained in the thawing stage were concentrated by the block technique to recover the retained solids. Each technique was individually studied. Based on the results, an integration of the techniques in a global process was proposed.

2.2.1. Falling film freeze concentration tests

The FFFC tests were developed in a multi-plate freeze concentrator shown in Fig. 2(a). The equipment included a freezing chamber, a freezing system, and a hydraulic system. The freezing unit consisted of two cooling plates with dimensions of 0.8 m width and 0.6 m height in a closed chamber. The hydraulic system spreads the coffee extract by means of two distributors with several holes 3 mm in diameter. The coffee extract flows in a descending film over the cooling plates, and it was collected in a collector tank and recirculated by a centrifugal pump. The ice growth on the surface of the freezing plates was then removed in a batch operation.

The freezing system consisted of refrigeration cycle using the primary refrigerant R-507 with a compressor (Tecumseh Europe,

La Verpilliere, France), a condenser and an expansion valve. The evaporation of the refrigerant occurred in the interior of the cooling plates, transferring the energy through the walls of the plate. Consequently, this process produces layer crystallisation in which the ice forms in thin layers on the surface of the heat exchanger.

All the stages of FFFC started with 40 kg of coffee extract. The stage ended when the ice achieved an average width of 25–35 mm. After that, the ice was removed, and the concentrated liquid fraction was used in the next stage. The initial mass in the next stage was obtained adding extract prepared at the concentration of the extract used in the previous stage. A total of seven stages of FFFC were developed. The mass of the concentrated fractions and the ice was measured on a PS 60-KB scale with 1 g precision (Gram Precision, Spain). The solid concentration percentage (Cs) was measured by refractometry (Atago Pal 100, Japan). The relationship between Brix degrees and Cs is represented by the equation $CS = 0.87^{\circ}\text{Brix}$ reported by Moreno et al. (2014) for the same coffee used in the present study.

2.2.2. Coffee solids recovery by partial thawing tests

Thawing tests were performed according to the method described by Gulfo et al. (2013). Cylindrical samples with a diameter of 60 mm, thickness from 25 to 35 mm and weight between 65 and 75 g were taken from the ice obtained at each of the seven stages of FFFC to study the solute recovery for fractionated thawing. A drill equipped with a puncher (Esgarret, Spain) was used to obtain six samples homogeneously distributed in each of the six ice sheets, for a total of 36 samples. Samples were taken in a refrigerated chamber to avoid melting the ice.

The samples were subjected to thawing tests in the setup shown in Fig. 2(b). The setup consisted of a cubic isolated chamber with 0.48 m sides. The chamber has a temperature control system (Pie Electro Dit, model 11 551, 0–300 W) and a 4-channel data logger (Testo 177-T4, Germany). The thawing was carried out at 20 ± 1 °C in a vertical position, similar to the position that the ice layers had in the freeze concentrator. The dripping of the melting ice is collected by a funnel connected to a container on a scale (Ohaus PA3102, USA) with a precision of 0.01 g to measure the mass. Ten thawing fractions of equal mass were separated, and the solid concentration was measured by refractometry. The average concentration was calculated from the data of the six samples of ice from each stage.

2.2.3. Block freeze concentration tests

Based on the results of the fractionated thawing tests, the diluted fractions of this stage (fractions where the concentration index was less than 1) were mixed and freeze-concentrated by the total block technique. The conditions of the FC test were as follows: cooling temperature –10 °C, thawing temperature 20 °C and thawing direction opposite to the freezing direction according to the best results reported by Moreno et al. (2014).

The block freeze concentrator is shown in Fig. 2(c). One hundred sixty grams of the coffee sample was placed into a cylindrical double jacketed container measuring 5.2 cm in diameter and 8.5 cm in height. The heat exchange fluid was a mixture of ethylene glycol

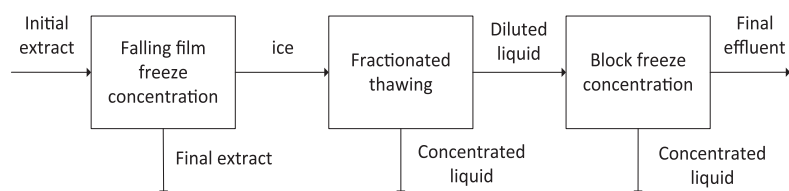


Fig. 1. Flowchart of freeze concentration tests.

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