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# Centrifugal freeze concentration

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ABSTRACT

Assisted techniques that improve the efficiency of freeze concentration in one-step configurations are important in achieving commercial viability. An alternative reported in this study is the use of the centrifugation for separating the concentrated solution from the ice matrix. By applying centrifugation, high values of solute had recovered, reaching approximately 0.73 kg of sucrose obtained per 1 kg of initial sucrose at 1600 RCF of centrifugation speed, independent of initial concentration of sucrose (5 to 20 wt.%) and freezing procedure (radial or unidirectional freezing). The performance of centrifugal freeze concentration may be attributed to ice matrix that acted as a porous solid through which the concentrated solution percolates through drainage channels between ice crystals improved by the centrifugal force.

*Industrial Relevance:* The freeze concentration allows producing food concentrates with high quality as compared to evaporation and membrane technology. In this work, centrifugation has been applied as an assisted technique to improve the efficiency of freeze concentration in one-step configuration, obtaining promissory results.



## 1. Introduction

Freeze concentration is a method for recovering a food solute from a solution based on the separation of pure ice crystals from a freeze-concentrated aqueous phase. As compared to evaporation and membrane technology, freeze concentration has some significant potential advantages for producing a concentrate with high quality because the process occurs at low temperatures where no vapor/liquid interface exists resulting in minimal loss of volatiles. Thus, the flavor and quality of freeze-concentrated products are exceptionally high, especially relative to their counterparts produced by evaporation. These benefits make freeze concentration particularly suitable for the concentration of some products, such as fruit juices, coffee and tea extracts, and aroma extracts (Morison & Hartel, 2007).

Recently, the industrial future of freeze concentration has been associated more with developments in the configuration of one-step systems (block freeze concentration or progressive freeze concentration) than conventional freeze concentration systems (suspension crystallization), because of the simpler separation step (Miyawaki, Kato, & Watabe, 2012; Petzold & Aguilera, 2009; Sánchez, Ruiz, Auleda, Hernández, & Raventós, 2009; Sánchez, Ruiz, Raventós, Auleda, & Hernández, 2010). An additional advantage of these one-step systems is their simplicity in terms of both the construction and operation of the equipment (Sánchez et al., 2009). In block freeze concentration, the whole liquid food is frozen (ice block), then thawed and the concentrated fraction is separated from the ice fraction by gravitational means. Under these conditions the control of thawing process becomes critical to achieve an efficiency of near 90% (Aider & de Halleux, 2008, 2009; Aider, de Halleux, & Melnikova, 2008). Progressive freeze concentration is based on a similar concept because a large ice mass is formed and grown on the cooling surface so that the separation from the mother solution is relatively easy (Liu, Miyawaki, & Nakamura, 1997).

Assisted techniques that improve the efficiency of processing in one-step configurations of freeze concentration are important in achieving commercial viability. Examples of the assisted technique are the use of ultrasound to control ice nucleation in progressive freeze concentration (Kawasaki, Matsuda, & Shiraishi, 2004; Matsuda & Kawasaki, 1997; Matsuda, Kawasaki, & Kadota, 1999) and the use of ice nucleation agents in progressive freeze concentration to avoid impurities in the ice phase to suppress the initial supercooling (Liu et al., 1997). Other alternatives are the use of external forces such as vacuum or centrifugation. In this way, vacuum (suction by a pump) has been proposed by Hsieh (2008) to obtain drinkable water from sea water to separate salt, thereby converting the ice of sea water into fresh water. Recently, Petzold, Niranjan, and Aguilera (2013) applying a vacuum (80 kPa) improved the efficiency over atmospheric conditions in freeze concentration of sucrose solutions. Centrifugation has been proposed by Bonilla-Zavaleta, Vernon-Carter, and Beristain (2006) in frozen pineapple juice to separate ice from unfrozen concentrated juice, while Luo, Chen, and Han (2010) obtained ice crystals of high purity during the freezing concentration of brackish water, and recently Virgen-Ortíz et al. (2012) proposed a simple

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and effective cryoconcentration method that elutes the concentrated protein solution from the frozen ice matrix via centrifugation.

Centrifugation is a type of separation where the force of gravity is largely replaced by a higher driving force, through the application of centrifugal force (Toledo, 2007). Thus, an alternative for separating the concentrated solution from the ice fraction is the use of centrifugation. The process takes advantage of the hydraulic system existing in the frozen matrix formed by veins (or channels) between the ice crystals occluding the concentrated solution. This matrix in a frozen system is responsible for differences in the concentration of impurities in ancient polar ice, where solutes migrated through the microchannels between the ice crystals under the pressure of upper ice layers (Rempel, Waddington, Wettaufer, & Worster, 2001).

The aim of this study was to analyze the use of centrifugation as a driving force to remove the concentrated solution from the ice matrix in a one-step freeze concentration of sucrose solutions.

## 2. Materials and methods

### 2.1. Materials

Aqueous solutions of sucrose (Sigma-Aldrich, Dorset, UK) with concentrations 5, 10, 15 and 20 wt.% were prepared with distilled water for assays.

### 2.2. Experimental procedure

#### 2.2.1. General experimental procedure

A general experimental procedure is schematized in Fig. 1. Sucrose solutions were frozen separately by radial and unidirectional freezing, and then the samples were transferred to a refrigerated centrifuge to force the separation of solutes from the frozen solutions.

#### 2.2.2. Freezing and centrifugation

Sucrose solutions (12 mL) contained in plastic centrifugal tubes (internal diameter D = 15 mm) were frozen in a static freezer at −20 °C for 12 h. As shown in Fig. 2, the external surface of the plastic tubes was covered with a thermal insulation made of foamed polystyrene (8 mm thickness, thermal conductivity K = 0.035 W $m^{-1}$ $K^{-1}$) so that heat transfer during freezing mainly occurred either unidirectionally (axially from top to bottom) (Fig. 2a) or radially from walls to the center (Fig. 2b). During freezing, the temperature in the sample was measured using needle-type copper-constantan thermocouples (Ellab A/S, Rodovre, Denmark) located at the geometric center of each of three test samples. Thermocouples were connected to a data acquisition system model CTF84-S8 (Ellab A/S, Copenhagen, Denmark) and registered continuously. The freezing rate (°C $min^{-1}$) was calculated as the rate of temperature decrease from the freezing temperature to the temperature 10 °C below the freezing point (Ramaswamy & Marcotte, 2006).

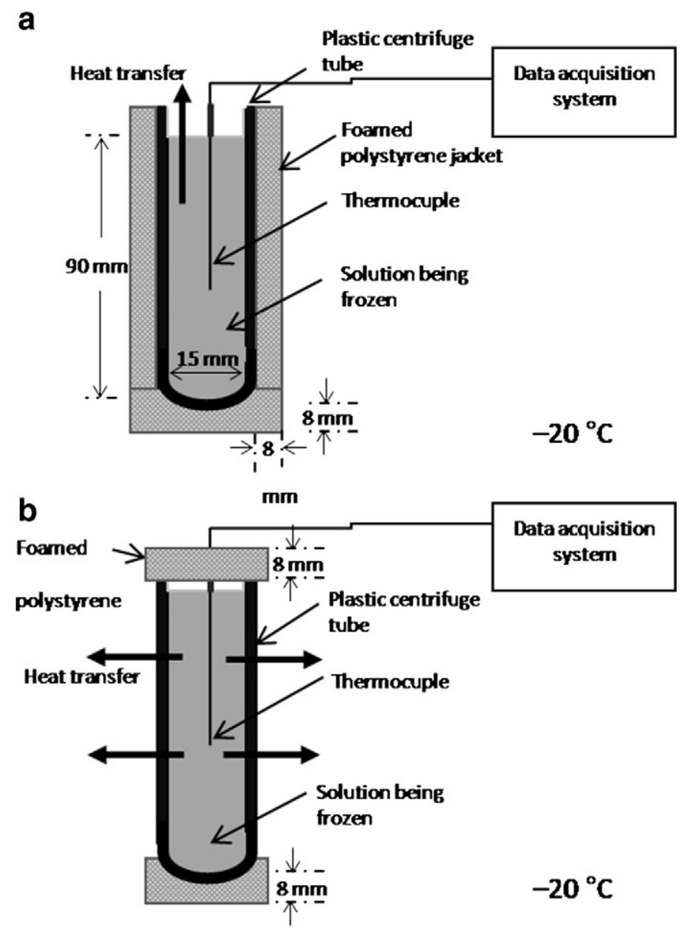


**Fig. 2.** Freezing conditions of samples. The samples were frozen in a static freezer at −20 °C for 12 h, where the heat transfer mainly occurs unidirectionally from top to bottom (a), and mainly radially from walls to the center (b).

The frozen samples were removed from the freezer and rapidly transferred to a refrigerated centrifuge (centrifuge Hettich model D-7853, Tuttlingen, Germany) operated at 20 °C for 15 min to force the separation of solutes from the frozen samples by different centrifugation speeds, expressed as a relative centrifuge force (RCF), see Fig. 1.

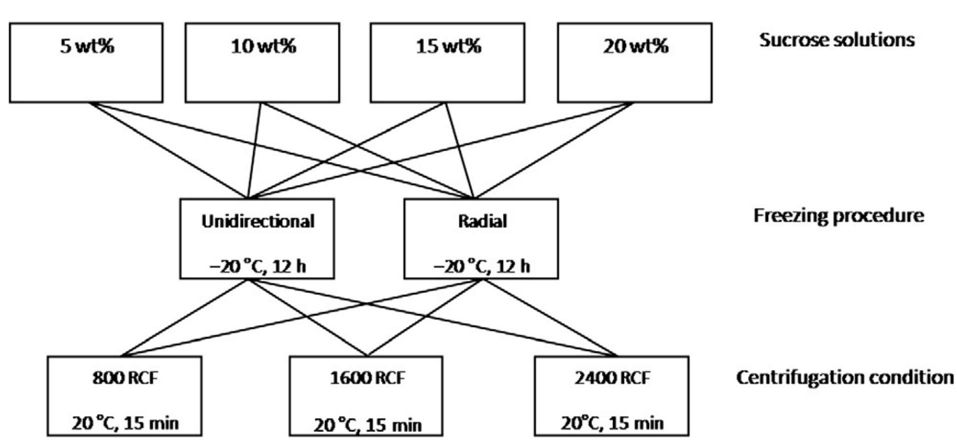


**Fig. 1.** General experimental procedure. Sucrose samples were freezing unidirectional and radial, and then transferred to a refrigerated centrifuge to force the separation of solutes from the frozen solutions.