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Calculation process for the recovery of solutes retained in the ice in a multi-plate freeze concentrator: Time and concentration

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ABSTRACT

Fractionated thawing was studied as a method to recover solutes incorporated in the ice obtained in a cryoconcentrator. Thawing times and solute concentration in the ice were determined at several thawing temperatures.

Ice sheets used for the thawing studies were obtained by cryoconcentrated solutions of sugars and simulated juice at initial concentrations of 5 to 20 °Brix. The ice sheets produced contained levels of solutes between 1.0 and 9.0 °Brix.

Fractionated thawing was performed at temperatures of 20 to 30 °C while maintaining geometrical similarity for the test samples. By fractionated thawing more than 60% of the solutes retained in the ice was recovered in 34%, of the total thawing time.

The procedure presented allows the determination of the solute concentration achieved in the various thawing fractions and predicts the thawing time required for a given form factor, melting temperature and the solute mass fraction in the ice.

Industrial relevance: The freeze concentration is a technology that allows eliminating water from liquid foods at temperatures below the water's freezing point, which allows obtaining products of better quality. Fractionated thawing was studied as a method to recover solutes incorporated in the ice, improving the global efficiency of a freeze concentration process by optimal recovery of the solutes retained in the ice. It also provides estimations of the energy used for thawing. This work continues the research in falling film freeze concentration technology which we have submitted and published in this journal. This work contributes to increase the global process efficiency.

1. Introduction

Freeze concentration is a method to separate soluble solids from an aqueous solution. It is based on the gradual lowering of the temperature of the solution until partial or total freezing is obtained. The advantage of this process as emerging technology for food processing is that it preserves the sensorial and nutritional quality of the food product. The low operational temperatures prevent degradation and denaturation reactions that can occur for concentration processes at elevated temperature, such as evaporation (Hartel & Chung, 1992).

According to literature (Chen & Chen, 2000; Chen, Chen, & Free, 1998; Chen, Chen, & Free, 1999; Flesland, 1995; Gunathilake, Shimmura, & Miyawaki, 2013; Hernández, Raventós, Auleda, & Ibarz, 2009; Miyawaki, 2001; Müller & Sekoulov, 1992; Sánchez, Ruiz, Hernández, Auleda, & Raventos, 2010; Yee, Wakisaka, Shirai, & Hassan, 2003), there are various basic methods for freeze concentration in solutions: suspension freeze concentration, layer freeze concentration, and block freeze concentration (or freeze–thaw concentration). These names originate from the mechanisms used for the formation of the ice crystals in the process.

Freeze concentration processes used industrially are currently all based on suspension freeze concentration. This process uses scraped-surface heat exchangers (SSHE) to generate ice nuclei, recrystalisers to increase the growth of the ice crystals and pressurised wash columns to separate the ice from the concentrated liquid. The concentration reached with this system is between 45 and 55 °Brix (Van Weelden, 1994; Lee & Lee, 1998). To reduce SSHE costs Otero, Sanz, Guignon, & Sanz (2012) suggest increasing the pressure at the nucleation stage.

Block freeze concentration, or freeze–thaw concentration, consists of the freezing of the product that is to be concentrated (liquid foods) and thawing while dividing the obtained liquid in various fractions: the first fractions are found to be concentrated (corresponding to high solute mass fractions in the ice) and the later fractions are found to be diluted (Boaventura et al., 2013; Miyawaki, Kato, & Watabe, 2012; Moreno, Raventós, Hernández, & Ruiz, 2014; Nakagawa, Maebashi, & Maeda, 2009; Yee et al., 2003).

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Nomenclature

a	length of ice layer (m)
b	width of ice layer (m)
b_{ni}	variable to correct the non-ideal behaviour
b_2	time constant (s^{-1})
e	thickness of ice layers (m)
f	fructose
ff	Form factor (m^{-1})
g	glucose
k	heat conductivity ($W \cdot m^{-1} \cdot °C^{-1}$)
p	weight sample (kg)
Peach (s)	simulated peach juice
r	coefficient in the linear correlation of Pearson
s	saccharose
t_{ct}	time (minutes)
v_{des}	thawing rate (mL/s)
AI	Accumulated index
AT	Accumulated time (minutes)
ºBx	concentration (°Brix)
CI	concentration index
C_{e1}	specific heat for carbohydrates ($kJ \cdot kg^{-1} \cdot ºC^{-1}$)
C_{e2}	specific heat for ice ($kJ \cdot kg^{-1} \cdot ºC^{-1}$)
FPD	freezing point depression (°C)
HPLC	high-performance liquid chromatography
K	coefficient of thawing ($s \cdot m^2 \cdot kg^{-1}$)
K_c	freezing point depression for water ($°C \cdot kg/mol$)
IL	ice layers
M	mass (kg)
M_s	molecular weights (g/mol)
PS	process stage of freeze concentration
PFC	progressive freeze-concentration
R	radius (m)
RMS	root mean square (%)
SSHE	scraped-surface heat exchangers
S	surface area (m^2)
SMFI	solute mass fraction in the ice (°Brix)
TS	test sample
TT	thawing time (min)
T	temperature (°C)
T_{CI}	temperature of the ice with retained solutes (°C)
T_{amb}	ambient temperature (°C)
T_{ats}	average temperature of the sample (−18 °C)
T_{ref}	cold store temperature (−18 °C)
$T_{thawing}$	thawing temperature (°C)
V	Volume (m^3)
X_c	mass fraction of solute (kg/kg)
X_{ice}	mass fraction of ice (kg/kg)
X_t	total mass fraction (kg/kg)

Greek symbols

α	individual heat transfer coefficient for heat transfer by convection (film coefficient) ($Wm^{-2} \cdot °C^{-1}$)
β	inverse of the time constant (s^{-1})
ρ_c	density of carbohydrates as function of temperature (kg/m^3)
ρ_{ice}	density of ice as function of temperature (kg/m^3)
ρ_{CI}	density of ice (−4.0 °C) (kg/m^3)
ρ_{-18}	density of ice (−18 °C) (kg/m^3)
$\Theta_{1\text{-}2}$	dimensionless number

Layer freeze concentration is based on a large ice mass formed and grown on the cooling surface. Two processes of layer freeze concentration have been described: progressive freeze-concentration (PFC), with ice formation on a tubular or cylindrical surface (Miyawaki et al., 2012) and multi-plate freeze-concentration, with ice formation on a rectangular plate, normally in the form of a falling-film freeze concentrator, where the liquid to be concentrated flows down in a layer over the heat-exchanger plates.

A characteristic of the falling-film freeze concentration process is the limitation in the maximum obtainable concentration, which is about 40% (w/w). This is principally due to the increase in viscosity of the liquid, the freezing-point depression, and the incorporation of solutes in the ice crystals (Chen et al., 1998; Flesland, 1995; Miyawaki, 2001; Raventós, Hernández, Auleda, & Ibarz, 2007; Sánchez, Hernández, Auleda, & Raventos, 2011).

At decreasing temperatures, the liquid reduces its flow velocity over the plate surface, which results in increased solute retention in the ice, and reduced process efficiency (Flesland, 1995; Raventós et al., 2007).

Scholz (1993) described the process of solute inclusion in a growing ice layer as a sequence of three individual phases: nucleation, the growth rate of the ice layer, and the final concentration obtained in the process. The retention of solutes in the ice sheet limits the applicability of freeze concentration falling film. This system seeks to recover these solutes and increase the overall efficiency of the process. A possible way to recover the solutes incorporated in the ice layer is fractionated thawing. Fractionated thawing is a complimentary technique which can be used in the process of falling-film freeze concentration. It consists of slowly heating the ice-layer obtained from the freeze concentrator and collecting the liquid that is formed in various fractions. A number of publications (Miyawaki et al., 2012; Yee et al., 2003) indicate that highly concentrated solutions can be obtained based on fractionated thawing.

The aim of the current paper is to study fractionated thawing as a method to recover the solutes incorporated in the ice layer formed in a falling-film freeze concentrator.

Thawing times are determined at various temperatures and solute concentrations. Moreover, a calculation algorithm is developed that predicts the concentration index and the time needed to obtain a certain mass fraction of thawing for solutions of fructose, glucose and saccharose at temperatures of 20, 25 and 30 °C.

This type of study is not only of academic interest, but has also technological importance, because it enables the calculation of the recovery of solutes retained in the ice obtained from fruit juices. Process parameters that are obtained include the thawing time, the concentration and amount of ice obtained in each of the thawing fractions of the process.

Based on these calculations, one can study the solute recovery in the various fractions, so that an optimal process can be designed.

2. Materials and methods

2.1. Sugars and model solution for peach juice and orange juice

- Fructose (f): D(−) fructose, crystalline white, 99%, (natural sugar from fruit, DietRádissonPagesa, Spain).
- Glucose (g): D + glucose anhydrous, extra pure, Ph, Eur, USP, BP (GL01251000, synthetic product, Scharlau, Spain).
- Saccharose (s): commercial crystalline white sugar; (a-D-glucopiranosil β-D-fructofuranòsid; saccharose, cane sugar, Azucarera, Spain).

Model solutions for peach juice were prepared, based on the weight percentages (w/w) of basic sugars (f, g, and s) obtained from HPLC analysis of peach juice Auleda, Raventós, Sánchez, and Hernández (2011),