CHEMICAL ENGINEERING*TRANSACTIONS*

VOL. 57, 2017

A publication of
AIDIC
The Italian Association
of Chemical Engineering
Online at www.aidic.it/cet

Guest Editors: Sauro Pierucci, Jiří JaromírKlemeš, Laura Piazza, SerafimBakalis

ISBN978-88-95608- 48-8; **ISSN** 2283-9216

DOI: 10.3303/CET1757299

Process Parameters of Vacuum-assisted Freeze Concentration

Guillermo Petzold, Patricio Orellana, Jorge Moreno, Carolina Cuevas

Group of Emergent Technology and Bioactive Components of Food, Department of Food Engineering, Universidad del Bío-Bío, Av. Andrés Bello s/n, Casilla 447, Chillán, Chile
gpetzold@ubiobio.cl

Freeze concentration is an effective unit operation to concentrate liquid foods such as fruit juices without the use of high temperatures. Recently, the innovations of freeze concentration have been associated more with the one-step configurations than conventional freeze concentration systems (suspension crystallization), because of the simpler separation step. Assisted techniques that improve the process parameters of freeze concentration in one-step are important in achieving commercial viability. The vacuum (suction by a pump) is an interesting assisted technique applied to freeze concentration in a one step. The objective was to study the process parameters of vacuum-assisted freeze concentration applied to orange juice. As material raw, using orange var. Navel and the juice were filtered to separate solids that might interfere with the cryoconcentration process. We used a vacuum pump at 80 kPa as an assisted technique to force the separation of concentrate (solutes) from the frozen solution at controlled temperature condition (20 °C). Over the time, under vacuum condition, the solids content (expressed as °Brix) in the concentrated fraction increased significantly compared the original sample, showing a kinetic of the solute elution from the ice block. The results show an evident advantage using the vacuum as an assisted technique compared the atmospheric condition. In addition, the efficiency under vacuum achieved high values over 70%, with high values of ice purity. The freeze concentration process using vacuum is similar to the principle used by children to suck the sugar solution with attractive colorants from popsicles and takes advantage of the frozen matrix formed by veins (or channels) between the ice crystals containing the concentrated solution. Under these conditions, the frozen block (the ice) acts as a carcass through which the concentrated fraction (rich in solids) passes, collecting the concentrate by gravitation force. The vacuum-assisted freeze concentration is an effective technique to obtain an orange fruit juice concentrated using a system in a one step with advantages compared the conventional suspension crystallization, which needs several steps and an important equipment capital inversion.

1. Introduction

The concentration of liquid food is an important unit operation because concentrated products occupy less space and weight (Ramaswamy and Marcotte, 2006) and the consumers only add water to obtain a high-quality final product. In this context, the concentration of fruit juices confers some economic advantages in packaging, storage, and distribution, for example in the case of orange juice. In the recent years, consumer demand for high-quality fruit juices had led to searches for emerging food technologies such as freeze concentration for liquid food concentration (Sandhu and Minhas, 2006). Freeze concentration is an interesting technology to minimize the loss of valuable components in the preparation of a liquid food concentrate, specifically fruit juices (Sánchez et al., 2009).

Freeze concentration is a technology where an aqueous food solution is concentrated via partial water freezing and separating the ice fraction from the concentrated residual solution (Aider and Ounis, 2012). Compared to evaporation and membrane technology, freeze concentration has some significant potential advantages for producing a high-quality liquid food concentrate because the low temperatures used in the process result in a minimal loss of volatiles (Morison and Hartel, 2007; Raventós et al., 2012). In addition, freeze concentration is an effective technology for protecting the valuable heat-labile components of liquid foods, as noted by Petzold et al. (2016a).

Please cite this article as: Petzold G., Orellana P., Moreno J., Cuevas C., 2017, Process parameters of vacuum-assisted freeze concentration, Chemical Engineering Transactions, 57, 1789-1794 DOI: 10.3303/CET1757299

Innovations of freeze concentration have been associated more commonly with developments in the configuration of one-step systems (block freeze concentration or progressive freeze concentration) than with conventional freeze concentration systems (suspension crystallization) because of the simpler separation step (Petzold and Aguilera, 2009; Miyawaki et al., 2012). Another 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, a liquid food solution is completely frozen, the whole frozen solution is thawed, and then the concentrated fraction is separated from the ice by gravitational thawing, sometimes assisted by other techniques to enhance efficiency (Aider and de Halleux, 2008). Under these conditions, the ice block acts as a solid carcass through which the concentrated fraction passes (Aider and de Halleux, 2009).

Assisted techniques that improve the process parameters of block freeze concentration in one-step are important in achieving commercial viability of this technology. Alternatives to assisted techniques applied to block freeze concentration include the use of external forces such as centrifugation or vacuum. In this way, centrifugation was proposed by Bonilla-Zavaleta et al. (2006) for the concentration of frozen pineapple juice, whereas Petzold and Aguilera (2013) presented a centrifugal freeze concentration method using a sucrose solution, and recently, Petzold et al. (2015) proposed the application of this assisted technique to fruit juices. Vacuum (suction by a pump) has been proposed by Hsieh (2008) to get drinkable water from seawater. On the other hand, Petzold et al. (2013) applying a vacuum suction improved the efficiency over atmospheric conditions in freeze concentration of sucrose solutions, and Moreno et al. (2013) reported the positive effect of vacuum on the movement of the concentrated liquid fraction in block freeze concentration of coffee brews. Pardo and Sánchez (2015) used a vacuum to the intensification of block freeze concentration applied of sucrose solutions. Recently, Petzold et al. (2016b) proposed the application of this assisted technique to block freeze concentration of red wine.

In this condition, freeze concentration assisted by an external force (such as vacuum) is similar to the principle used by children to suck the sugar solution containing colorants from popsicles, takes advantage of the hydraulic system that exists in the frozen matrix between the ice crystals occluding the solutes (Petzold et al., 2013). A similar behaviour is potentially observed in nature in that this frozen system is responsible for the differences in the concentration of impurities in ancient polar ice (Rempel et al., 2001).

The aim of this paper was to study the process parameters of vacuum-assisted freeze concentration applied to orange juice.

2. Material and Methods

2.1 Materials

Oranges var. Navel were obtained from commercial sources (Chillán, Chile) and were kept under refrigeration (5 °C, overnight) until processing. Oranges were squeezed, and the juice was filtered to separate out the seeds and solids that might interfere with the cryoconcentration process. Following this process, orange juice was ready for experimental use.

2.2 Freezing and vacuum suction procedure

The freezing conditions were performed by the method of Petzold and Aguilera (2013), with slight modifications. Orange juice (45 ml) was placed in plastic tubes (internal diameter = 22 mm) and was frozen at -20 ºC for 12 h in a static freezer. For the freezing, the external surface of the plastic tubes were covered with a thermal insulation made of foamed polystyrene (8 mm thickness), so that the heat transfer occurred unidirectional (axis from bottom to top). During freezing, a needle-type copper-constantan thermocouple (Ellab A/S, Rodovre, Denmark) was inserted in the geometric center of the samples to record continuously the temperature of the sample with a data acquisition system model CTF84-S8 (Ellab A/S, Copenhagen, Denmark). The freezing rate (μm/s) was calculated as the distance divided by the freezing time (based on the ice propagation rate) (Ramaswamy and Marcotte, 2006).

To force the separation of concentrate with solutes from the ice matrix, the frozen juice was rapidly removed from the freezer and transferred to a suction stage (Petzold et al. 2016b). The suction was generated by connecting a vacuum pump (80 kPa, Medi-pump 1636; Thomas Ind., WI, USA) to the bottom of the frozen sample at controlled temperature using a refrigerated incubator (20 °C ± 1, FOC 215E; Velp Scientific Inc., Milano, Italy). The vacuum was controlled visually with a vacuum manometer of the pump and an external manometer. Separately, the same procedure was performed but using only gravity to force the separation of solutes from the frozen sample. Over the time (120 min) under vacuum and atmospheric conditions, initial and final weights of the ice fraction were registered, the concentrated solution was recovered and the frozen fraction was thawed, and thus, concentrations of solids were determined in both fractions.

2.3 Process parameters

The concentration of fractions C_f and C_s (solids in the molten frozen phase and concentrated solution, respectively) obtained over the time was analysed at ambient temperature (approximately 22 °C) with a digital refractometer (PAL-1, Atago Inc., Japan), with a precision of ± 0.1 °Brix.

The efficiency over time was calculated using the following equation:

$$\eta(\%) = \left(\frac{C_S - C_f}{C_S} \right) \times 100 \tag{1}$$

Where C_s and C_f are the concentration of solids (°Brix) in the concentrated solution and frozen fraction, respectively.

2.4 Validation of results

To validate the obtained experimental results, a mass balance was made and compared to theoretical value as follows:

$$W_p = \frac{C_S - C_0}{C_S - C_f} \tag{2}$$

Where C_0 is the initial concentration of solids; and Wp is the predicted value of ice mass ratio W (kg ice/kg initial).

The quality of the fit between experimental (We) and predicted (Wp) values for N experimental points over time were tested by the root mean square (RMS) as follows:

$$\mathrm{RMS}(\%) = 100 \sqrt{\frac{\sum \left[{(W_e - W_p)}/{W_e} \right]^2}{N}} \tag{3}$$

2.5 Statistical analysis

Statistical analysis of data was performed through analysis of variance (ANOVA) using Statgraphics Centurion XVI Software (Statgraphics, 2009). Differences among mean values were established by the least significant difference (LSD) at 5%.

3. Results and discussion

3.1 Process parameters

Figure 1 shows the solids content (ºBrix) of the concentrated (C_s) and ice fraction (C_f) as a function of the time of the process, under vacuum (Figure 1a) and atmospheric condition (Figure 1b).

Figure 1a shows the first times under vacuum that the solids content reached a value near 47 ºBrix (20 min), which was the highest value and represented an increase of 4.5 times the solids content of the fresh sample. However, by increasing the time under vacuum, the solids in the concentrate decreased progressively due to a normal kinetic suction of the solids from a frozen solution matrix. In this condition, at the final stage of the suction process (120 min), the concentrate had higher solids content (19 ºBrix) compared to the fresh juice, with high ice purity (6 ºBrix) in the frozen remaining fraction. A similar behaviour of the kinetic suction was observed using frozen wine (Petzold et al., 2016b).

On the other hand, using atmospheric condition (Figure 1b) shows an evident less satisfactory concentration effect compared to the vacuum process, reaching values near of 23 ºBrix at 20 min, decreasing progressively the concentration of solids in the concentrate until reaching a value close to 14 ºBrix at 120 min. These results are in agreement with the comparative studies between vacuum and gravity using sucrose by Petzold et al. (2013).

The efficiency was greater with the vacuum condition than with the atmospheric condition (Figure 2). Under vacuum condition the efficiency achieved high values over 70% and with the atmospheric condition only reaching values of efficiency near about 60%, thus demonstrating the advantage of using vacuum as an assisting technique for freeze concentration. However, these results using vacuum showed a lower efficiency when compared with Petzold et al. (2013), since obtained 86% of efficiency by applying vacuum for 20 min in a sucrose solution (C_0 = 10 ºBrix) concentrated by block freeze concentration, a difference attributed to the more complex composition of the orange juice compared with a sucrose solution that could be more complicated the separation process.

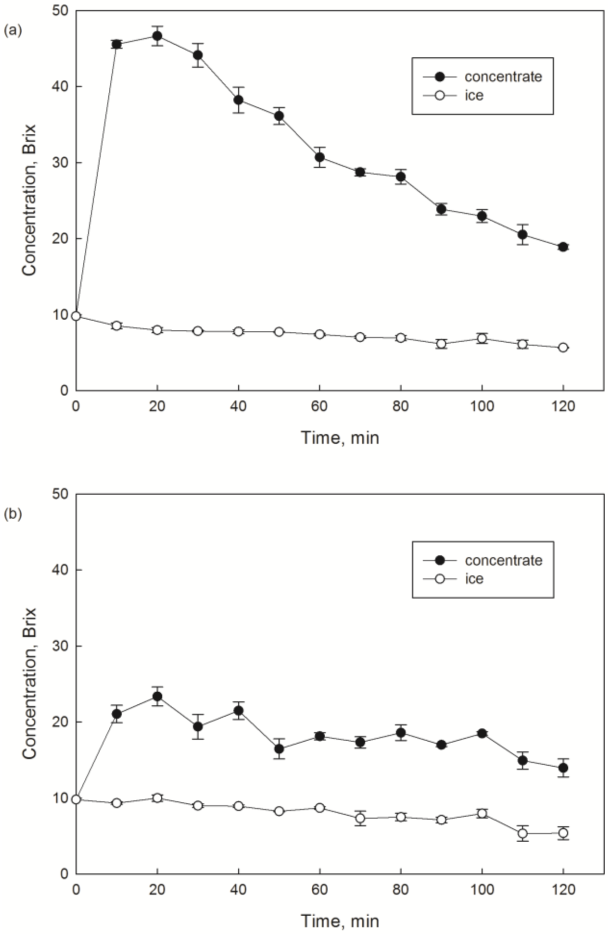

Figure 1: Solids content (ºBrix) of the concentrated (C_s) and ice fraction (C_f) as a function of the time, under vacuum (a) and atmospheric condition (b).

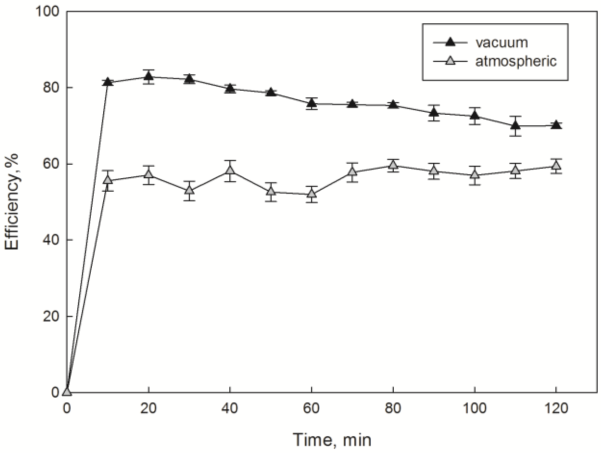

Figure 2: Evolution of efficiency as a function of the time, under vacuum and atmospheric conditions.

Another important factor in the block freeze concentration performance is the freezing rate of the liquid food sample. In this case, orange juice has a moderate freezing rate of 5.9 μm/s (Ramaswamy and Marcotte, 2006), which is lower than the critical value (approximately 8 μm/s) reported by Nakagawa et al. (2010) and Moreno et al. (2014). These authors reported that for velocities higher than 8 μm/s, the ice occluded solutes during the freezing stage and is not possible to expect a considerable separation of the concentrated solution.

3.2 Validation of results

To validate the experimental results, a mass balance was made over time of the process (Eq. 2). The ice mass ratio (W) presented a linear downward trend (Figure 3). A good agreement was observed between the experimental (We) and predicted (Wp) ice mass ratios for vacuum (Figure 3a) and atmospheric condition (Figure 3b). The obtained RMS (Eq. 3) values for vacuum and atmospheric treatments were 8.2% and 9.3%, respectively. These values were lower than 25%, which Lewicki (2000) considered being an acceptable fit, and these values were close to those reported in the literature (Hernández et al., 2010; Petzold et al., 2013, 2016b).

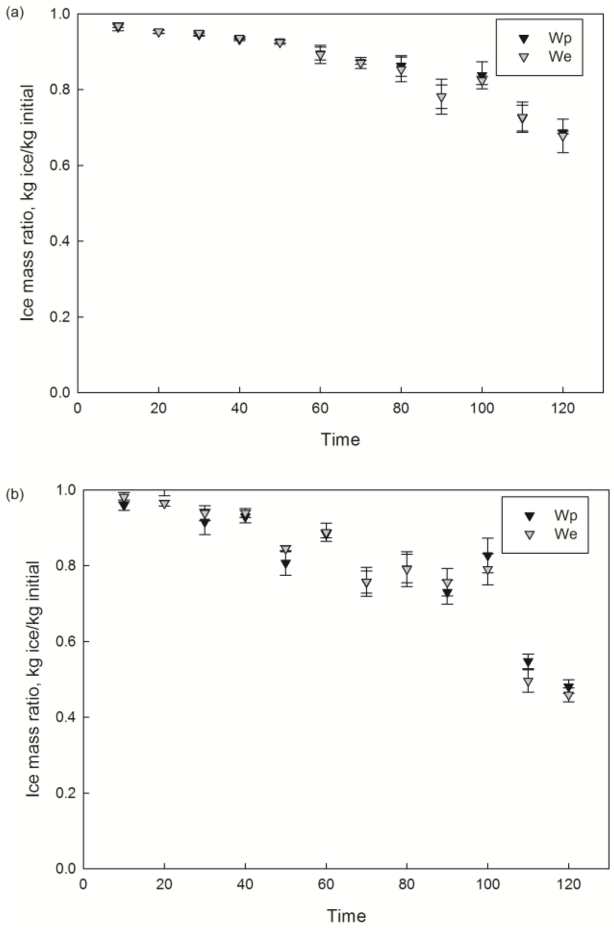

Figure 3: Experimental (We) and predicted (Wp) ice mass ratios as a function of the time, under vacuum (a) and atmospheric condition (b).

4. Conclusions

Vacuum used as an assisted technique in freeze concentration of orange juice showed evident advantages compared to the atmospheric treatment. Vacuum improved the process parameters, increased 4.5 times the initial solids content in the first times of suction, and achieved values over 70% for efficiency and high values of ice purity. From a practical point of view, vacuum-assisted freeze concentration technique can be considered as an excellent tool to elaborate a concentrate from a food liquid such as orange juice.

Acknowledgments

Author Guillermo Petzold is grateful for the financial support provided by CONICYT through FONDECYT Project No. 11140747

References

Aider M., de Halleux D., 2008. Production of concentrated cherry and apricot juices by cryoconcentration technology. LWT-Food Sci. Technol., 41, 1768–1775.

Aider M., de Halleux D., 2009. Cryoconcentration technology in the bio-food industry: principles and applications. LWT-Food Sci. Technol., 42, 679–685.

Aider M., Ounis W.B., 2012. Skim milk cryoconcentration as affected by the thawing mode: gravitational vs. microwave-assisted. Int. J. Food Sci. Technol., 47, 195–202.

Bonilla-Zavaleta E., Vernon-Carter E.J., Beristain C.I., 2006. Thermophysical properties of freeze-concentrated pineapple juice. Ital. J. Food Sci., 18, 367–376.

Hernández E., Raventós M., Auleda J.M., Ibarz A., 2010. Freeze concentration of must in a pilot plant falling film cryoconcentrator. Innov. Food Sci. Emerg. Technol., 11, 130–136.

Hsieh H.-C., 2008. Desalinating process. US Patent, 7,467,526.

Lewicki P.P., 2000. Raoult's law based food water sorption isotherm. J. Food Eng., 43, 31–40.

Miyawaki O., Kato S., Watabe K., 2012. Yield improvement in progressive freeze concentration by partial melting of ice. J. Food Eng., 108, 377–382.

Moreno F.L., Robles C.M., Sarmiento Z., Ruiz Y., Pardo J.M., 2013. Effect of separation and thawing mode on block freeze-concentration of coffee brews. Food Bioprod. Process., 91, 396–402.

Moreno F.L., Raventós M., Hernández E., Ruiz Y., 2014. Block freeze-concentration of coffee extract: Effect of freezing and thawing stages on solute recovery and bioactive compounds. J. Food Eng., 120, 158–166.

Morison K.R. Hartel R.W., 2007. Evaporation and freeze concentration, In Heldman D.R. and Lund D.B. (eds), Handbook of Food Engineering. CRC Press, New York, USA, 495-552.

Nakagawa K., Maebashi S., Maeda K., 2010. Freezing and thawing as a path to concentrate aqueous solution. Sep. Purif. Technol., 73, 403–408.

Pardo M., Sánchez R., 2015. Block freeze concentration intensification by means of vacuum and microwave pulses. Ingenieria y Competitividad, 17, 143–151.

Petzold G., Aguilera J.M., 2009. Ice morphology: fundamentals and technological applications in foods. Food Biophys., 4, 378–396.

Petzold G., Aguilera J.M., 2013. Centrifugal freeze concentration.Innov. Food Sci. Emerg., 20, 253–258.

Petzold G., Moreno J., Lastra P., Rojas K., Orellana P., 2015. Block freeze concentration assisted by centrifugation applied to blueberry and pineapple juices. Innov. Food Sci. Emerg., 30, 192–197.

Petzold G., Niranjan K., Aguilera J.M., 2013. Vacuum-assisted freeze concentration of sucrose solutions. J. Food Eng., 115, 357–361.

Petzold G., Orellana P., Moreno J., Junod J., Bugueño G., 2016a. Freeze concentration as a technique to protect valuable heat-labile components of foods, In J.J. Moreno (ed.), Innovative processing technologies for foods with bioactive compounds. CRC Press, Boca Raton, Florida, USA, 184-190.

Petzold G., Orellana P., Moreno J., Cerda E., Parra P., 2016b. Vacuum-assisted block freeze concentration applied to wine. Innov. Food Sci. Emerg., 36, 330–335.

Ramaswamy H., Marcotte M., 2006. Food processing. Principles and applications. Taylor & Francis, Boca Raton, Florida, USA.

Raventós M., Hernández E., Auleda J.M., 2012. Freeze Concentration Applications in Fruit Processing, In S. Rodríguez, F.A.N. Fernandes (eds.), Advances in Fruit Processing Technologies. CRC Press, Boca Raton, Florida, USA, 263–286.

Rempel A.W., Waddington E.D., Wettaufer J.S., Worster M.G., 2001. Possible displacement of the climate signal in ancient ice by premelting and anomalous diffusion. Nature, 411, 568–571.

Sánchez J., Ruiz Y., Auleda J. M., Hernández E., Raventós M., 2009. Review. Freeze concentration in the fruit juices industry. Food Sci. Technol. Int., 15, 303–315.

Sandhu K.S., Minhas K.S., 2006. Oranges and Citrus Juices, In Y.H. Hui (ed.), Handbook of Fruits and Fruit Processing. Blackwell Publishing, Iowa, USA.309.

Statgraphics, 2009. Statgraphics centurion XVI.StatPoint Technologies, Inc.,Warrenton, Virgina, USA.