Progressive freeze concentration of orange juice in a pilot plant falling film

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A B S T R A C T

In the present study the cryoconcentration of orange juice was investigated, using a pilot plant unit for cryoconcentration of liquids using a cold surface. The evolution in time of the concentration of solids in the juice and in the ice was analyzed during the process. It was found that the concentration of solids in the juice showed a linear increase in time at a rate of 0.75 °Brix/h until a final concentration of 28.8 °Brix. The concentration of solutes in the ice showed an exponential increase.

Industrial relevance: The freeze concentration allows dewatering orange juice at temperatures below the water’s freezing point, which allows obtaining products of better quality. The freeze concentration is a technology that allows eliminating water from the juices at temperatures below the water’s freezing point, which allows obtaining products of better quality. This work has applied this technology to concentrate orange juice, obtaining promissory results.

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1. Introduction

Freeze concentration of liquid foods is a technology that minimises losses of volatiles and thermolabile components. This renders a final product with a quality that cannot be obtained by conventional concentration processes used in the food industry, such as evaporation and membrane concentration. Cryoconcentration is a technique for concentrating liquid products by means of freezing and the subsequent separation of part of the frozen water from the liquid product. The process involves lowering the temperature of the product to be concentrated to below its freezing point in a controlled manner in order to avoid reaching the eutectic temperature at which all the components of the product solidify at once. If it reaches the eutectic point of the juice, separation is very difficult. The aim is to obtain a very pure ice, i.e. only water, without retaining any of the solids in the product. The purpose of removing this ice is to obtain a concentrated liquid product.

Concentration by freezing is the system that comes closest to the ideal objective of separating water from the food product without affecting the other components. The greatest advantages offered by the use of cryoconcentration rather than other technologies are the low temperatures reached in the process and the non-existence of a liquid–vapour interface. There is no loss of volatiles, making this technique very suitable for the concentration of thermo sensitive fluids (Fellows, 2007).

There are many studies available on the application of this technique to the concentration of kiwi juice (Maltini & Mastrocola, 1999), berry juices (Chizzoni, Del Popolo, & Porretta, 1995; Di Cesare, Nani, Brambilla, Tessari, & Fussari, 2000), apple and pear juices (Nazir & Farid, 2008; Hernández, Raventós, Auleda, & Ibarz, 2009), but where it is most commonly applied is to citrus fruits juice (Braddock & Marcy, 1987; van Nistelrooij, 2005). This concentration technique has also been applied in other food industries, such as dairy products (Hartel & Espinel, 1993; van Nistelrooij, 2005), brewing (Putman, Vanderhasselt, & Vanhamel, 1997), winery and distilling (Cesare, Cortesi, & Maltini, 1993), and for the concentration of dilute solutions of tea and coffee.

According to various researchers (Flesland, 1995; Chen, Chen, & Free, 1998; Miyawaki, 2001; Aider & de Halleux, 2009), there are two basic methods for freeze concentration of liquid foods. In the conventional method of suspension crystallization, individual ice crystals are formed that are enlarged in size by Ostwald ripening. Separation between ice crystals and concentrate is quite complicated in a pressurized wash column. The second method (progressive freeze concentration) is the crystallization of water, forming an ice layer present in the solution in the form of an ice layer on a cold surface. Large ice mass is formed and grown on the cooling surface and the separation from concentrate mother liquor is relatively easy.

Although most current systems of freeze concentration work with suspension crystallization, it is suggested in recent studies (Sánchez, Ruiz, Auleda, Hernández, & Raventós, 2009; Petzold & Aguilera, 2009; Auleda, Hernández, & Raventós, 2009) that the future of freeze concentration applications seems to be associated more with developments in the configuration of progressive freeze concentration systems than advances in suspension crystallization, because of the simpler separation step.
The freeze concentration equipment designed at the food industry pilot plant of the Technical University of Catalonia is a type of progressive freeze concentration, based on freezing the water content of fluids in direct contact with a cold surface. A layer of ice is formed on the exchange surface, made up of stainless steel plates through which a refrigerant fluid circulates. The fluid to be concentrated is circulated through the system using a pump. The system successfully concentrated sugar solutions (Raventós, Hernández, Auleda, & Ibarz, 2007), apple and pear juices (Hernández et al., 2009), and must (Hernández, Raventós, Auleda, & Ibarz, 2010) obtaining concentrations up to 30 °Brix. It was showed that this kind of progressive freeze concentration equipment could be of interest as a pre-concentration system in the food industry.

The principal aim of the present paper is to study the process of freeze concentration of orange juice using a multi-plate device. To do so we will examine the behavior of certain variables that can define the efficiency of this type of concentration. Average concentration rate, relative impurity of ice, and energy consumption were analyzed. The concentration limits by this method were also explored.

2. Materials and methods

2.1. Experimental set-up

The equipment comprises a freezer unit, a freezing system, a hydraulic system and an electric system (Fig. 1). The hydraulic system circulates the fluid to be concentrated from the holding tank to the freezer unit, where it is chilled, and then returns it to the tank to be circulated again. The freezing system freezes the fluid to be concentrated, forming ice in the freezer unit. The system used to remove the ice, i.e. defrosting, is not mechanical, but instead is a reversal of the refrigeration cycle. When defrosting, the ice is melted and detaches from the plates, falling into a tray.

The crystallization unit is an indirect contact freezer, where the energy for the refrigeration is transferred through the walls of a plate heat exchanger. Consequently, this produces layer crystallization in which the ice forms in thin layers on the surface of the heat exchanger (800 mm × 600 mm).

The compressor is the model TFH4531Z by Unité Hermétique (Tecumseh Europe, La Verpilliere, France), of the piston alternative volumetric type, with single effect, one stage, and with vertical cylinders, since the motor shaft and the crankshaft of the compressor are on the same axis. The model used in this experiment was a hermetic compressor run by a three-phase 230/400 V, 50 Hz. Using the primary refrigerant R-507, the caloric power or output of the compressor will depend on the evaporation and condensation temperature of the refrigerant fluid.

The freezer consists of five evaporator plates laid out vertically, each one formed in turn by two welded plates. Inside, the plates are welded at several points so that the refrigerant R-507 circulates through them following a zigzag path. Each of the five plates measures

![Fig. 1. Basic scheme of experimental freezing unit.](image-url)
800 mm × 600 mm. On the outside of the plates, the liquid to be concentrated flows in a descending film after being pumped from the tank and distributed by means of six distributors with small-diameter perforations. An exchange of heat occurs between the evaporating refrigerant and the liquid to be chilled. Note that, when defrosting, the evaporator plates perform the function of condensers.

The liquid flow is measured by an electromagnetic flow meter ISOIL Model MS501 (Cinisello Balsamo, Italy) of stainless steel for fluids with conductivity over 5 μS/cm. The measurement range is 0.14–3.47 L/s with a precision of 0.4%.

2.2. Sample preparation

The orange juice used in this study (200 L) was a pasteurised and clarified juice with a concentration of 11.1 °Brix, obtained from a juice processor in the area of Lérida, Spain. The sample was held at 0 °C until the start of the tests.

2.3. Sugar content

The levels of fructose, glucose and sucrose in orange juice were determined using High Performance Liquid Chromatography (HPLC, Beckman, San Ramon (CA)), with a Spherisorb NH2 (25 × 0.4 cm) column, 5 micro particles; mobile phase: acetonitrile: water (75:25); flow rate: 1.5 mL/min; injection volume: 20 μL. The analyses were repeated three times.

2.4. Concentration measurement (°Brix)

The concentration of soluble solids present in fruit pulp and juice is usually expressed in °Brix, which is equivalent to the percentage of sucrose. The concentration of soluble solids was measured using an ATAGO refractometer (model DBX-55A) with a precision of ±0.1 °Brix.

2.5. Freeze point

The freeze point of (concentrated) orange juice was determined as a function of the concentration of soluble solids. For this, 10 mL samples of juices obtained in the same processor with concentrations of 10.1, 15, 19.2, 24.6, 29.8, 35.4, 40.5 and 46.2 °Brix were used. The samples were put in a freezer at −30 °C. Temperatures were registered every 15 s using a K Thermopar sensor TESTO model 177-T4 (TESTO, Germany), with accuracy 0.1 °C, previously calibrated with distilled water and placed in the middle of the tube. Data were collected using a data logger, connected to a PC. In this way we obtained the freezing curves of orange juices at the different concentrations of soluble solids.

The lowest temperature of the freezing curve shows the starting process of ice crystal formation (nucleation). It is followed by a temperature increase due to latent heat of the phase change (crystallization). The highest temperature reached at that point corresponds to the freezing point (Tfp) of the sample.

2.6. Freeze concentration tests

Freeze concentration tests were performed using thirty-five liters (35 L) of orange juice with an initial concentration of 11.1 °Brix. During the tests, a layer of ice was formed on the plates, which became thicker as the equipment continued working. Based on previous tests carried out using the same equipment (Raventós et al., 2007), the optimum thickness for the ice on the plates was known to be approximately 10 mm. For the tests with orange juice it was initially planned to concentrate the juice in various steps. However, in the tests using orange juice, it was noticed that a large amount of foam was formed that obstructed the movement and distribution of the fluid. This reduced the contact area between the juice and the plates and thus reduced the heat transfer. Moreover, the foam made the freeze concentration equipment difficult to handle. To avoid foaming it was decided not to limit the duration of the concentration stages by attaining a given thickness of ice. Instead the duration of the tests was determined by a constant average flow of 1.1 ± 0.2 L/s sufficient to ensure good contact between the plate and the fluid being concentrated. Four concentration stages were used for the orange juice concentration. During each of these stages measurements were taken for concentration, temperature of the juice, evaporation temperature, and flow rate at different times. The weight of the separated ice was measured at the end of each stage, as was the content of soluble solids of both the separated ice and the juices. All the ice formed was melted to obtain a representative result for the concentration of solids in °Brix.

The quantity of water which must be removed from a liquid to raise the solids concentration from the initial value to the desired final value is given by Eq. (1) (Burdo, Kovalenko, & Kharenko, 2008):

\[
W_{\text{prene}} = \frac{C_{\text{fsl}} - C_{\text{f}}}{C_{\text{fsl}} - C_{\text{ice}}}
\]

where:

\[
W_{\text{prene}} = \text{predicted ice mass ratio (kg ice/initial kg juice)}
\]

\[
C_{\text{f}} = \text{initial concentration of juice (°Brix)}
\]

\[
C_{\text{fsl}} = \text{final concentration of juice (°Brix)}
\]

\[
C_{\text{ice}} = \text{solids concentration of ice (°Brix)}
\]

2.7. Ice growth rate

The kinetics of the juice concentration is directly related to the growth rate of the ice crystals on the wall, which is determined by heat and mass transfer (Hartel, 1992; Caretta, Courtot, & Davies, 2006). The heat transfer resistance is conductive because the heat of crystallization is transported through the ice to the wall (Pronk, 2006). When heat transfer is the limiting factor, the ice growth rate can be described by Eq. (2):

\[
\nu_{\text{ice}} = \frac{\lambda_{\text{ice}}}{\theta_{\text{ice}} \delta_{\text{ice}} \Delta T_{i-f}}
\]

where:

\[
\nu_{\text{ice}} = \text{linear growth rate of ice (m/s)}
\]

\[
\theta_{\text{ice}} = \text{thermal conductivity of ice (W/m °C)}
\]

\[
\delta_{\text{ice}} = \text{average thickness of the ice layer formed (m)}
\]

The temperature at the interface (T) can be estimated by Fourier's law at the end of each test using the thermal power by the equipment (Pt) at the evaporation temperature (Tw), the mean thickness of the ice layer and the effective surface area of heat transfer.

It is known that in processes of directional ice growth a phenomenon of concentration polarization occurs in the ice–solution interface, with accumulation of solutes close to the ice that is formed (Nagashima & Furukawa, 1997; Butler, 2002). These solutes tend to move by diffusion towards the less concentrated solution. The ice mass transfer resistance of ice growth in aqueous solutions (food liquids) can be estimated by Eq. (3) (Huige & Thijssen, 1972; Hartel, 1992):

\[
\nu_{\text{ice}} = \left(\frac{\kappa_{\text{sol}} \rho_{\text{i}}}{C_{\text{i}} \rho_{\text{ice}}} \right) \left(\frac{\delta_{\text{ice}}}{\Delta T_{i-f}} \right)
\]

where:

\[
\nu_{\text{ice}} = \text{linear growth rate of ice (m/s)}
\]

\[
\kappa_{\text{sol}} = \text{mass transfer coefficient (m/s)}
\]
The interface concentration $C_i$ is calculated from the freezing point using (Eq. (8)), and the mass transfer coefficient ($k_{m}$) is estimated based on dimensionless numbers (Re, Sc, Sh) as described by Flesland (1995) in a paper on freeze concentration by layer crystallization in a falling film.

The mean ice growth rate during each process step of freeze concentration can be estimated by Eq. (4) (Chen, Chen, & Free, 1999).

$$ V_{\text{ice}} = \frac{M \cdot (1 - C_{\text{ice}})}{A \cdot T_{\text{ice}}} $$  \hspace{1cm} (4)

where $C_{\text{ice}}$ is the ice concentration (°Brix), $A$ is the area covered by ice on the plate surface (m²), $V_{\text{ice}}$ is the average ice growth rate (m/s); $\rho_{\text{ice}}$ is the density of pure ice (kg/m³); $\alpha$ is the time needed for ice growth (s); $M$ is the mass of melted ice-water (kg).

The physical properties of ice and orange juice, needed for these calculations, were obtained from correlations given in Table 1.

Where:

$$ \eta $: juice viscosity (mPa s)

$C$: concentration soluble solids (°Brix)

$T$: temperature (K) (Ibarz et al., 2009) or (°C) (Hernández, 2008; Choi & Okos, 1986)

$\rho_{j}$: juice density (kg/m³)

$\rho_{\text{ice}}$: ice density (kg/m³)

$\lambda_{\text{ice}}$: thermal conductivity of ice (W/m °C)

$\lambda_{\text{juice}}$: thermal conductivity of pure ice (W/m °C)

$\lambda_{\text{HC}}$: thermal conductivity of carbohydrate (W/m °C)

$\chi_{\text{ice}}$: volumetric fraction of ice

$\chi_{\text{juice}}$: volumetric fraction of carbohydrate

$D$: diffusion coefficient (m²/s)

$T_{\text{fp}}$: freezing point (°C)

2.8. Relative impurity of the ice

The impurity of the ice was determined as the concentration of soluble solids (measured in °Brix) from the liquid phase that remains in the ice after freezing and therefore reduces its purity. The relative impurity of the ice is defined as the ratio between the concentration in the ice and the concentration in the concentrated juice at the end of each test:

$$ \text{Impurity ratio(%) = } \frac{C_{\text{ice}}}{C_{\text{juice}}} \times 100 $$  \hspace{1cm} (5)

where $C_{\text{juice}}$ is the concentration of soluble solids in the solution and $C_{\text{ice}}$ is the concentration of soluble solids in the ice at the end of each experiment.

2.9. Statistical analysis

The experimental results obtained from this study were fitted to different mathematical models using SAS statistical package (SAS Institute Inc. NC, USA, version 8). The fits and estimates were calculated at a significance level of 95%.

3. Results and discussion

3.1. Fruit juice characterization

The concentrations of glucose, fructose and sucrose in the orange juice were determined by HPLC. The proportions of sugars in juice at initial concentration of 11.1 °Brix are: glucose 29 ± 0.4%, fructose 27 ± 0.4% and sucrose 44 ± 0.4%. In the clarified and depectinized orange juice, sugar composition determines the rheological behavior of the tested juice. Viscosity is one of the most decisive properties for the flow juice over the walls of the freezer plates.

3.2. Mass balance

In order to validate the obtained experimental results a mass balance of each test was made, which was compared with theory (Eq. (1)).

The ice mass ratio has a downward trend with the concentration. The ice mass ratio varies with the difference of concentration between the liquid phase and ice. The solubles of the liquid phase drop linearly, while the ice phase concentration grows exponentially during cryoconcentration process (Raventós et al., 2007; Hernández et al., 2009). Thus the concentration difference is increased over the process and reduces the ice mass ratio. The exponential trend of solutes retained in the ice is closely related to the viscosity of the juice, which for Newtonian fluids also shows an exponential dependence (Arrhenius type) with the concentration and temperature. The composition of the concentrated juice sugar (sucrose, glucose and fructose) has a significant influence on the viscosity.

The viscosity of sugars solutions when compared the same temperature and concentration, increased in the following order: fructose, glucose and sucrose (Telis, Telís-Romero, Mazzotti, & Gabas, 2007; Raventós et al., 2007). To find out how they influence the composition requires further experimentation.

Fig. 2 compares the experimental results with theoretical predictions by Eq. (1) for each test. A good agreement was observed between ice mass ratio predicted by Eq. (1) and the one obtained experimentally. The quality of the fit was evaluated by the root mean square (RMS) given by:

$$ \text{RMS(%) = } 100 \sqrt{\frac{\sum (W_{\text{exp}} - W_{\text{pred}})^2}{W_{\text{exp}}^2}} $$  \hspace{1cm} (6)

where $W_{\text{exp}}$ and $W_{\text{pred}}$ are, respectively, the experimental and predicted ice mass ratio, and $N$ is the number of available experimental points. The RMS value obtained is 5.2%, much lower than the 25% that Lewicki (2000) considers for acceptance of a fit.

3.3. Freezing point ($T_{\text{fp}}$)

Freezing points depend on the concentration and type of soluble solids. The freezing point values for orange juice at different concentrations of soluble solids are shown in Fig. 3. The freezing point values for orange juice dropped as concentration of soluble solids increased. The sugar composition of orange juice shows that for the same concentration the freezing point of the juice reached
intermediate values between the freezing points of sucrose and monosaccharides (Raventós et al., 2007).

Fitting the experimental data of $T_{fp}$ with a second order polynomial results in Eqs. (7) and (8), valid between 10.1 and 46.2 °Brix:

$$
T_{fp} = -0.0094C^2 + 0.203C - 2.786 \quad (R^2 = 0.986) \tag{7}
$$

$$
C = -0.228T_{fp}^2 - 6.229T_{fp} + 3.428 \quad (R^2 = 0.997) \tag{8}
$$

where $T_{fp}$ is the freezing point of the orange juice, and $C$ is the concentration of soluble solids in the juice in °Brix.

### 3.4. Time dependence of concentration and amount of ice formed

During the freeze concentration process, the juice that is recirculated has a progressively higher content of soluble solids as the amount of ice eliminated increases. Fig. 4 shows the variation of the concentration of the juice and the weight of the ice eliminated over the processing time. It is noted that in both cases the changes in those two variables show a linear trend of the following type:

$$
y = b + ax \tag{9}
$$

where $y$ represents the concentration of the juice in °Brix or the weight of the ice in kg and $x$ represents the processing time in hours.

Using the experimental data shown in Fig. 4, a least squares fit was made with for which both the fit and the estimates of the parameters are significant with 95% probability. Table 2 shows the data for the fit parameters.

A linear trend is observed for the increase in concentration in time for orange juice (Fig. 4). A final concentration of 28.8 °Brix is reached.

In the paper by Bayindirli, Ozilgen, and Urgan (1993), a sigmoidal curve was fitted to describe the freeze concentration process of apple juice (between 12 and 40 °Brix) both for a single-step process and for a multi-step process. Similar results are found in the paper by Nonthanum and Tansakul (2008) with freeze concentration of lime juice between 7.6 and 17.3 °Brix. In these papers the concentration showed a phase of linear increase until a certain concentration value or a certain amount of ice is reached, after which the rate of increase drops off due to kinetic obstacles, which would represent the limits of the process.

It is thought that the results obtained in the present tests might be in the linear increase phase and that the limit of the process has probably not been reached. The value of 28.8 °Brix achieved in the last stage of freeze concentration is similar to the values of 26.6, 27.5 and 31.2 °Brix obtained using the same equipment with simple solutions of glucose, fructose and sucrose respectively (Raventós et al., 2007), and similar to the value of 29.5 obtained with must (Hernández et al., 2010). This may be due to the proportions of sugars in orange juice used in the tests (29% glucose, 27% fructose and 44% of sucrose). The kinetic coefficient for the tests with orange juice is 0.75 °Brix/h, while for the simple solutions of glucose, fructose and sucrose it was 1.19, 1.17, and 1.65 °Brix/h, respectively (Raventós et al., 2007). This difference may be caused by the lower heat transfer in the present tests with orange juice. Moreover, the formation of foam in the first steps of the process may have reduced the heat transfer, which may explain the lower effectivity of the process compared with the results obtained with simple sugar solutions.

The differing slopes for concentration and ice accumulation show that more than 1 kg of ice is needed to increase the concentration by 1 °Brix. Likewise, as occurs with all concentration systems, as the freeze concentration process advances, the amount of ice that must be eliminated to increase the concentration of the liquid phase by one unit is progressively smaller, which agrees with Eq. (1). That trend ranges from 2.8 kg of ice/°Brix during the second stage to 2.27 kg of ice/°Brix during the final stage. Since the cooling capacity of the equipment remains practically constant at an evaporation temperature of ~15 °C during all the tests, it would be desired to use a smaller heat-exchange surface as the juice is concentrated. If it would be

### Table 2

<table>
<thead>
<tr>
<th>Variable</th>
<th>$b$</th>
<th>$a$</th>
<th>$R^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Concentration</td>
<td>10.7 ± 0.16</td>
<td>0.75 ± 0.01</td>
<td>0.995</td>
</tr>
<tr>
<td>Accumulated ice</td>
<td>0</td>
<td>1.774 ± 0.025</td>
<td>0.998</td>
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</table>
possible to modify the heat-exchange surface of the equipment according to the stage of concentration, and one would probably obtain significant energy savings.

Although the general time-evolution of the concentration process is fitted well with a linear trend line (Fig. 4), a more detailed analysis for each process step indicates that the concentration increase slows down towards the end of each step, which is probably caused by kinetic limitations. Fig. 5 shows the juice concentration as a function of time during the fourth process step. It clearly shows an asymptotic tendency. It is of paramount importance to understand the phenomena that cause this tendency, both for understanding the limits of existing equipment and for progressing in the development of new equipment.

The kinetics of the juice concentration is directly related to the growth rate of the ice crystals on the wall, which is determined by heat and mass transfer (Huige & Thijsen, 1972; Hartel, 1992; Caretta et al., 2006).

Eqs. (2), (3), and (4) have been used to calculate the growth rate of ice during the tests with orange juice.

Fig. 6 shows the results obtained in steps 2, 3, and 4 of the test with cryoconcentration of orange juice.

The results suggest that at concentrations above about 20 °Brix the growth rate of ice is determined mainly by mass transfer. This can be compared with the results for sugar solutions obtained by Omran and King (1974) which indicate that mass transfer determines the growth rate of ice at concentrations above 15% w/w. Zhang and Hartel (1995) suggest that in the cryoconcentration of skim milk at concentrations between 10 and 17% w/w, mass transfer is not the limiting factor.

With respect to the limitations mentioned above, it would be possible to improve the equipment by replacing the current centrifugal pump by a positive displacement pump, thus avoiding vigorous agitation of the fluid and reducing foam formation. The mass transfer could be improved by increasing the flow of the juice. Note that the flow used in the present study (1.1 L/s) provides low Reynolds numbers (decreasing from 250 to 85). Flow can be increased by increasing diameter of the holes, and reducing the distance between the holes in the tubes that distribute the juice over the plates.

3.5. Evolution of impurity ratio

Fig. 7 shows the changes in the ratio of impurity of the ice during the freeze concentration process in relation with the concentration of the juice obtained. The experimental data obtained show a linear trend (Eq. (9)), although in this case the y variable represents the ratio of impurity and x is the concentration of the concentrated juice obtained at each stage, expressed in °Brix.

A least squares fit was made so that both the fit and the estimates of the parameters are significant with 95% probability. The results of the fit are shown in Table 3.

![Fig. 5. Time dependence of the juice concentration during the fourth process step.](image)

![Fig. 6. Ice growth rate during process steps 2, 3 and 4.](image)

![Fig. 7. Time dependence of the impurity ratio of the ice formed.](image)

The impurity ratio of the process increases with the increase in concentration because the solids retained in the ice increase with the concentration. It is believed that due to the increased viscosity of the solution with concentration and the decrease in temperature, the solutes accumulated in the interface due to concentration polarization have greater difficulty in moving towards the solution and are more easily retained in the ice being formed.

The levels of soluble solids in the ice formed (between 4.4 and 14.4 °Brix) could be related to the type of ice formed. Although microscopic observations of the ice were not performed, results from earlier studies with sugar solutions indicated the existence of a constitutional supercooling in the equipment used at concentrations of about 22 °Brix (Hernández, 2008). The consequence of this constitutional supercooling is the dendritic growth of ice, which enhances the retention of solutes (Ayel, Lottin, Fauch eux, Sallier, & Peerhossaini, 2006).

On the other hand, with regard to the purity of the ice formed, it is observed that as the concentration of the juice increases, the ice formed retains more solutes, following an exponential trend (Fig. 8).

In order to correlate that trend, the data shown in Fig. 8 have been fit to an exponential model, obtaining the following fit equation:

\[
C_{ic} = 1.35 \pm 0.39 \exp^{-0.082 \pm 0.011C_{JS}} \quad R^2 = 0.973
\]

where \( C_{ic} \) and \( C_{JS} \) are the concentration of soluble solids in the ice and in the concentrated juice, respectively, both expressed in °Brix. A similar trend has been observed in tests performed using the
same equipment with sugar solutions and must (Raventós et al., 2007; Hernández et al., 2009). In a recent paper of Ibarz et al. (2009) it is demonstrated that clarified and depectinized orange juice shows Newtonian behavior at temperatures near the freezing point. It is believed that the exponential trend in the purity of the ice formed is closely related to viscosity, since in Newtonian liquids viscosity shows an exponential trend with concentration (Ibarz & Pagán, 1987; Ibarz, Pagán, Gutiérrez, & Vicente, 1989).

3.6. Energy consumption

The energy consumption during the freeze concentration of orange juice is estimated taking into account the power rating of the equipment (2.75 kW at an evaporation temperature of −15 °C and a condensation temperature of +35 °C) and the duration of the different stages of freeze concentration. The energy consumption throughout the process varied between 1.4 kWh/kg of ice (5.1 MJ/kg of ice) during the second stage, and 1.6 kWh/kg of ice (5.9 MJ/kg of ice) during the last stage. This indicates that the equipment is more efficient at lower concentrations of the liquid phase. However, during the first step of the process the energy consumption was relatively large (1.8 kWh/kg of ice; 6.4 MJ/kg of ice), because of the formation of foam, that obstructed the heat transfer. The mean energy consumption between 11 and 28.8 °Brix is 1.5 kWh/kg of ice (5.4 MJ/kg of ice), which is less than the mean energy consumption indicated for the pilot plant of GEA Messo PT (based in suspension freeze concentration system) which is rated at 1.8 kWh/kg of ice (6.4 MJ/kg of ice) between 12 and 48 °Brix for citrus juice, using refrigerant R-507.

The energy consumption is higher than the level indicated in a review by Aider & de Halleux (2009), which is explained by the fact that the refrigeration equipment in the current study is based on a single-stage mechanical compression, without heat recovery. A way of reducing the energy consumption would be to reduce the surface area for heat exchange, following the decrease of the quantity of orange juice during the process.

4. Conclusions

Orange juice was successfully concentrated with a semi-industrial equipment for cryoconcentration, until a concentration of 28.8 °Brix. An average concentration rate of 0.75 °Brix/h was obtained and flow rates were around 1.1 ± 0.2 L/s. The estimated energy consumption increased from an initial 1.4 kWh/kg of ice to 1.6 kWh/kg of ice as the concentration of the fluid increased. The main limitations of the process at the concentrations used appear to be related to mass transfer.

5. Significance of the study

The freeze concentration allows dewatering orange juice at temperatures below the water’s freezing point, which allows obtaining products of better quality. This work has applied this technology to concentrate orange juice, obtaining promissory results.

Acknowledgements

We thank the juice industry manufacturer Nufri (Mollerusa-Spain) for supply of the juices used for testing. José Sánchez acknowledges the “Fondo Nacional de Ciencia y Tecnología” (FONACIT) of the Bolivarian Republic of Venezuela for a grant to perform his doctoral studies (2008–2012).

References


Table 3

<table>
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<tr>
<th>Variable</th>
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<th>a</th>
<th>R²</th>
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<td>Impurity ratio</td>
<td>0</td>
<td>1.82±0.10</td>
<td>0.834</td>
</tr>
</tbody>
</table>

Fig. 8. Relation between ice and juice concentration.


Van Nistelrooij, M. (2005). Bridging the cost barrier to freeze concentration. *Food and Beverage Asia, April/May.*