Performance of a Freeze Concentration Pilot Plant during the Start-Up Phase

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Abstract

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In this paper are presented the findings obtained during the start-up of a freeze concentration pilot plant, that operated with suspension crystallisation and with simulated fruit juices. Solutions with different initial concentrations were tested, with and without acidification, to simulate fruit characteristics and behaviour. The experimental runs demonstrated that solution acidity has negligible effects on the process performance, whereas the results showed that the initial solution concentration influences the start-up duration. During this time, the solution concentration grows by about 10 °Brix, and the maximum quantity of ice content inside the crystalliser is always lower than 40% by weight. These findings can help to predict the performance of a freeze concentration plant and may facilitate achieving target concentrations in a more efficient mode. Taken together, the data suggest that the initial concentration of the processed juice influences start-up duration and the achievable final concentration.

Keywords: crystalliser performance; crystal growth; crystal separation; ice content; suspension crystallization

The food industry currently finds itself in a phase characterised by a large number of innovative technological changes aimed at obtaining high-quality products and preserving the main organoleptic and nutritional characteristics.

In this context, freeze concentration represents a valid alternative to the traditional evaporation to separate water from a solution (Aider & De Halleux 2008; Aider & De Halleux 2009; Bremer Boaventura *et al*. 2013; Gunathilake *et al*. 2014; Miyawaki *et al*. 2016; Samsuri *et al*. 2016).

The concentration of fruit juices is done mainly for two reasons: (i) to improve preservation by increasing sugar content; (ii) to reduce storage and transport costs.

Evaporation has been always widely used due to the ease of its use; however, it has several drawbacks, mainly related to the loss of volatile components.

Freeze concentration is a technique that overcomes these drawbacks, preserving the organoleptic properties.

Until now, however, the technique has not been widely applied due to difficulties in controlling the whole process, which is based on three steps:

(1) crystal nucleation – ice nuclei are generated, resulting in a suspension of ice crystals in solution (slurry);

(2) crystal growth – crystal size increases to give large crystals that can be easily separated in the following step;

(3) crystal separation – the ice crystals are removed to recover the concentrated product.

The solutes and their concentrations affect the freezing point with a non-linear trend, as shown in previous studies (Chen *et al*. 1990; Chiampo & Conti 2000; Pradipasena *et al*. 2007; Gunathilake *et al*. 2013).

Determining freezing temperature is of vital importance for the whole process, since the ice crystals can grow only if the temperature is continuously decreased due to the increase in the concentration of the solution.

The most critical step is crystal growth. Larger and well-shaped crystals are separated from the slurry more easily than smaller ones, and for this reason the crystal growth step influences the crystal separation. Small crystals are lost in the concentrate itself leading to undesired re-dilution.

Two obstacles can hamper efficient ice separation: (1) product loss in the form of film adhered to the crystals, and (2) incomplete separation of small crystals. This means that ice separation efficiency is influenced by crystal washing and crystal growth.

This work presents the findings obtained in a freeze concentration pilot plant operated with simulated fruit juices during the plant start-up step and before crystal washing was commenced.

The results reported here represent a contribution to our knowledge regarding freeze concentration and indicate that the initial concentration of the processed juice influences the start-up duration and the achievable concentration.

MATERIAL AND METHODS

Pilot plant description. The pilot plant used for the experimental runs is sketched in Figure 1.

The fluid, stored in a refrigerated tank at a temperature close to its freezing point, is sent to a scraped surface cooler (flow rate = 35–60 l/h), where ice nucleation starts.

The cooler output feeds the crystalliser (volume = 0.12 m^3), where the nuclei can grow until they reach a size that is suitable for separation.

The crystalliser is jacketed and equipped with a screw stirrer, to maintain slurry homogeneity, both for temperature and for ice concentration. On its bottom there is a filter that blocks the passage of crystals over 152 μm: this allows large crystals to grow, while the smaller ones are recirculated to the cooler together with the concentrate. Samples are taken to analyse the solution concentration and the ice content. When the ice content is at least equal to 20% (the value depends on the initial concentration), the washing column can be switched on and part of the crystalliser content starts to be sent to this column. From this point onwards, the plant works in steady-state condition. This means that the ice content does not increase anymore and remains constant inside the crystalliser. When the steady-state condition starts, the washing column starts to work.

The washing column contains a piston, whose stainless-steel head has a series of slots and a central hole for the slurry feed (feeding occurs during downward piston movement). The upward movement allows the concentrate to leave the column through the piston head slots, while the ice crystals are compacted to form a bed that is washed by cold water to recover the concentrate adhered to the ice crystals.

The water used for washing is sent back to the cooler, while the clean ice bed is scraped by a device placed in the upper end of column, and sent to melting. This ice recovery system gives very low losses of concentrate (always lower than 0.1% by weight of solutes).

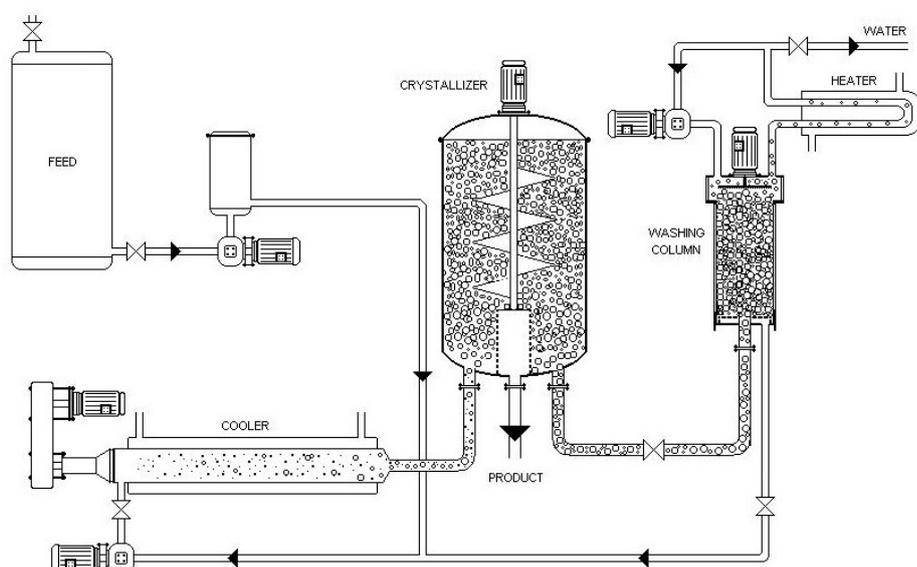The pilot plant is equipped to control and change its operative parameters.

Figure 1. Freeze concentration pilot plant

Table 1. Start-up duration

Initial solution concentration (°Brix)	12.8	11.0[a]	20.1	20.0[a]	29.1	30.3[a]	39.6
Start-up duration (h)	1.5	2.0	2.0	4.0	3.5	3.75	6.0

[a]acidified solution (pH = 3.5)

Analyses and measurements. During the experimental runs, the main parameters measured were

(1) the solution concentration (c_C) on samples taken at the crystalliser output and measured as Brix degrees by an ATAGO refractometer;

(2) the slurry concentration (c_S) measured as Brix degrees of the solution diluted by the ice melting (samples were taken directly from the crystalliser).

These parameters allow calculation of the ice content (*I*) (the percentage of the ice inside the slurry), defined as:

$$I = (1 - c_S / c_C) \times 100 \tag{1}$$

where: $c_C - c_S$ – the difference is the concentration of water in the form of ice inside the solution at a given time

Runs. The runs were carried out using sugar solutions to simulate fruit juices.

The main fruit sugars (fructose, glucose and sucrose) were used, and the mass ratio of fructose/glucose/sucrose was kept constant and was equal to 40/40/20. The initial concentration of the solution was in the range 11–39.6 °Brix. Some solutions were acidified with monohydrate citric acid (pH = 3.5) to determine the influence of pH.

During each run, solution concentration (c_C) and slurry concentration (c_S) were monitored and the ice content was calculated.

All the runs were carried out before operating the washing column (during the plant start-up).

RESULTS AND DISCUSSION

Figure 2A shows the concentration of solution inside the crystalliser during the start-up.

The first finding was that acidity had no relevant influence on increasing the concentration of the solution. However, start-up duration is influenced by the initial concentration of the solution (Table 1), which in turn influences the solution freezing point, since at the highest values mass transfer decreases due to the higher viscosity caused by the contemporaneous effects of lower temperatures and higher solute concentrations. The plot shows that the concentration growth rate decreases with the initial increase in concentration.

The runs also show that during the start-up the solution concentration grows by about 10 °Brix. On this basis, the growth period can be fitted with a linear plot whose slope gives the growth rate; the results are presented in Table 2.

Figure 2B shows the ice content during the start-up: the trend reproduces the results achieved for the tested solutions (as for the solution concentration, Table 2 shows the slopes of the fitting straight lines for the
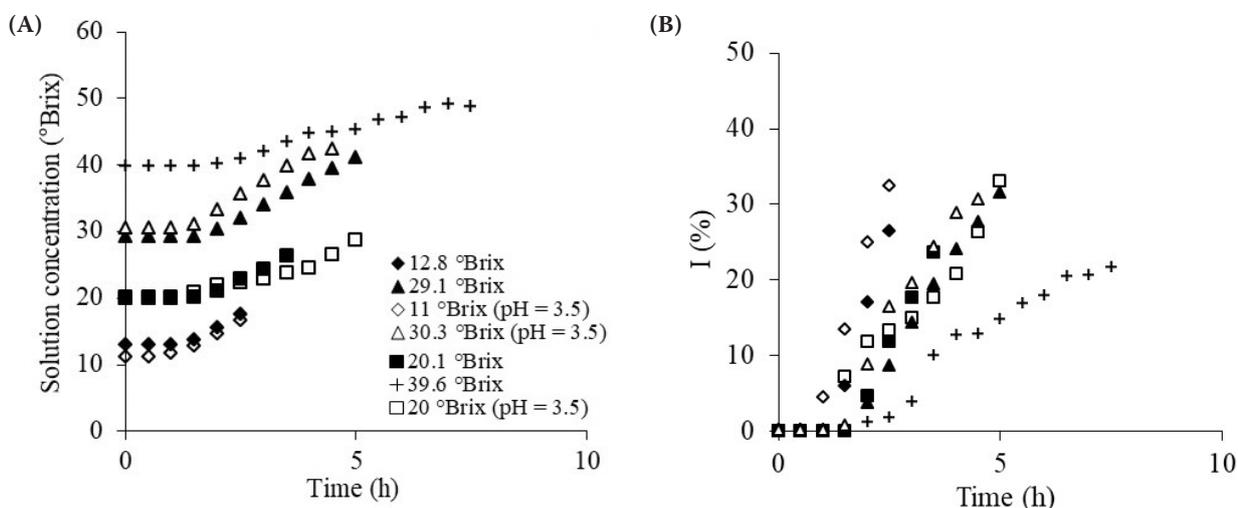

Figure 2. Monitoring of the solution concentration (**A**) and the crystallizer ice content (**B**) during the start-up period

Table 2. Growth rate of solution concentration and ice content

Initial solution concentration (°Brix)	Calculated growth rate		Removed/initial water	Ice production (kg/h)
	solution concentration (°Brix/h)	ice content (%/h)		
12.8	3.12	17.0	0.30	22.0
11.0[a]	2.76	15.7	0.36	20.2
20.1	3.14	11.7	0.38	19.9
20.0[a]	1.95	7.8	0.41	10.7
29.1	3.53	9.2	0.43	11.8
30.3[a]	3.77	9.3	0.44	11.0
39.6	1.68	3.9	0.36	5.0

[a]acidified solution (pH = 3.5)

growth period). The most striking finding is that the maximum values are always lower than 40% by weight.

Figure 3 presents the relationship between ice content and solution concentration:

(1) no appreciable differences were observed between acidified and non-acidified solutions;

(2) acidification does not influence the process; this result should be considered when acidic fruit juices are freeze-concentrated;

(3) the growth in ice content is influenced by the initial product concentration, with slower growth when the concentration of the solution is higher;

(4) the maximum ice content is always lower than 40% by weight.

The start-up duration is influenced by the mass transfer coefficient, which is in turn influenced by:

(1) solution viscosity: this parameter grows with the concentration of the solution and with decreasing temperature;

(2) diffusivity: this parameter has the same trend as viscosity.

Table 2. also reports the ratio of removed water/initial water and the ice production rate; for this latter parameter, the values decrease with increasing concentration of the solution.

CONCLUSIONS

The experimental runs demonstrated that the start-up duration of a freeze concentration pilot plant is strongly influenced by the initial solution concentration: this parameter controls mass transfer, ice crystal growth and operation rate. Solution acidity has negligible effects. Start-up duration increases with the initial concentration of the solution. During this period, the concentration can grow by about 10 °Brix, but, in any case, the ice concentration inside the crystallizer is always lower than 40% by weight. Although additional runs are required with real fruit juices, these findings constitute a contribution towards defining the operative parameters in similar processes on a larger scale.

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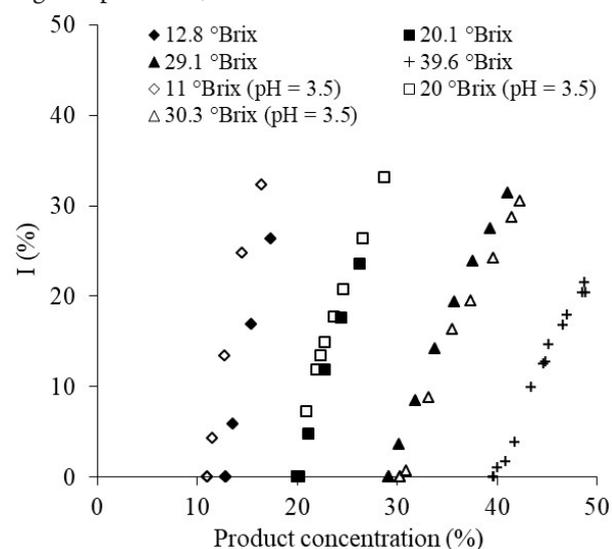
Figure 3. Effect of the initial solution concentration on crystallizer ice content

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