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Review: Freeze Concentration Technology Applied to Dairy Products

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Freeze concentration is a process of concentrating liquid products by freezing the water content and subsequently removing the so-formed ice crystals from the food system. In dairy processing, this technology offers the advantage of minimizing the heat abuse of sensitive milk components, such as proteins and flavors. It thus provides an opportunity for producing dairy ingredients with enhanced functional and organoleptic qualities. By freeze concentration, skim milk has been concentrated up to 40 wt% total solids (TS) and whole milk up to 44 wt% TS. Lactose and lipids are more concentrated in the ice fraction than in the concentrated fraction. Proteins (casein and whey protein) decrease the ice growth rate and the high viscosity is a limiting factor for the freeze concentration of both skim milk and whole milk. In this study, the most important studies relating to the suspension, block and layer freeze concentration of milk are summarized, analyzing results and indicating how freeze concentration process efficiency of dairy products can be improved.

Key Words: suspension crystallization, layer crystallization, block freeze concentration, skim milk, whole milk

INTRODUCTION

Evaporation, membrane concentration (reverse osmosis) and freeze concentration are three methods for the concentration of liquid foods. Evaporation uses gas–liquid phase separation. It has the lowest capital cost and highest maximum concentration obtainable (>50°Brix); yet, it provides the lowest product quality. Reverse osmosis is based on the molecular-sieve mechanism of semi-permeable membranes. The maximum concentration obtainable is relatively low (~30°Brix); it requires least energy for separation but the cost of membranes is high. Freeze concentration is based on solid–liquid phase separation at low temperatures so that a good retention of flavors and thermally sensitive components is expected. It gives the best quality and the total costs (including energy, capital and cleaning) are three to four times higher than for evaporation or reverse osmosis (van Mil and Bouman, 1990). The maximum concentration reached for freeze concentration is about 50°Brix (Heldman, 2003).

Because of the low temperatures used in freeze concentration, this technology can be an attractive alternative to the standard concentration techniques used in dairy processing at present, such as evaporation and membrane technologies. (Raventós et al., 2007; Habib and Farid, 2008; Hernández et al., 2009). Theoretically, when comparing the heat of evaporation (about 2260 kJ/kg under pressure of 0.1 MPa) with enthalpy of freezing (335 kJ/kg), the process of freeze concentration seems to be cheaper than evaporation from the energy point of view. On the other hand, due to the different forms of energy used (steam and electricity), it is appropriate to consider energy consumption expressed as primary energy. For example, if we consider 90% efficiency of boiler and 44% efficiency in electric production, the consumptions of primary energy per ton of water removed during whey concentration were: 45–90 MJ for reverse osmosis (between 5.5 and 11 wt%), 245–450 MJ for mechanical vapor recompression (MVR) (mechanical compression), 5-stage or 7-stage evaporation (between 5.5 and 53 wt%) and 490 MJ for freeze concentration (between 10 and 40 wt%; De Boer and Hiddink, 1980; van Mil and Bouman, 1990).

Sensory quality of freeze-concentrated milk has been reported to meet the quality of fresh skim milk and it exceeds those of conventionally concentrated and powdered skim milk when reconstituted as a beverage or when used as an ingredient in the development of cream cheese, frozen dessert, sour cream, salad

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dressings, whipped toppings and other products (Best and Vasavada, 1993).

Freeze concentration is a process for concentrating liquid products by freezing the water content and subsequently removing the so-formed ice crystals from the food system. According to Aider et al. (2009a), there are three basic mechanisms for ice crystal formation in solutions of dairy products. The first is known as suspension crystallization (Huige and Thijssen, 1972; Hartel and Espinel, 1993), consisting of an initial phase of ice nuclei formation (nucleation), also called crystallization, followed by a second phase which involves the growth of ice nuclei in the solution. The second method is the crystallization of water present in the solution in the form of an ice layer on a cold surface (layer crystallization) (Müller and Sekoulov, 1992; Flesland, 1995). The third method, known as block crystallization, occurs when a liquid solution is completely frozen and the temperature in the center of the product is largely below the freezing point. After that, the whole frozen solution is thawed and the concentrated fraction is separated from the ice fraction by means of gravitational thawing assisted or by other techniques to enhance the separation efficiency (Aider et al., 2008).

Based on these three mechanisms of ice crystallization, three techniques for freeze concentration have been developed, known as suspension freeze concentration, progressive freeze concentration and block freeze concentration, respectively.

The methods described for the formation of ice in solutions differ in terms of heat extraction (through ice layer or through solution). The ice growth rate is higher in progressive and block crystallization than in suspension crystallization; ice purity is higher in suspension crystallization and the equipment is more easy to use in progressive freeze concentration because there are no moving parts (Sanchez et al., 2009).

The experimental studies on freeze concentration of dairy products have resulted in an understanding of the

thermodynamics of the process, of the behavior of dairy fluids at low temperatures and of the influence of the main milk components (proteins, lactose and lipids) on the attainable concentration and on the purity of the ice.

The aim of this study is to summarize the most important studies relating to the freeze concentration of milk, to show and analyze the obtained results and to suggest how to improve process efficiency.

SUSPENSION FREEZE CONCENTRATION OF DAIRY PRODUCTS

The use of suspension freeze concentration has been studied experimentally for concentration of various dairy products. Most papers focus on the study of the process conditions that enable a fast growth rate of the ice crystals, in each case suggesting equations for the calculation of mass and heat transfer coefficients (Hartel and Espinel, 1993; Qin et al., 2003). In most studies, it is assumed that the rate of nucleation and growth of the crystals is directly related to the rate at which heat is removed and to the rate of mass diffusion (Hartel and Espinel, 1993; Zhang and Hartel, 1996). Two other lines of research, important for the application of cryoconcentration of dairy products, are the development of new equipment (Dickey et al., 1995; Zhang and Hartel, 1996; Habib and Farid, 2007) and the study of the influence of cryoconcentration on the proteins and lactose present in dairy products (Hartel and Chung, 1993).

Best and Vasavada (1993) report the freeze concentration of skim milk, whole milk, sweet whey, whey protein concentrate and whey permeate using a single-stage configured Niro pilot plant unit (Figure 1).

Maximum steady-state concentrations (expressed as concentration TS) achieved were 40 wt% for skim milk

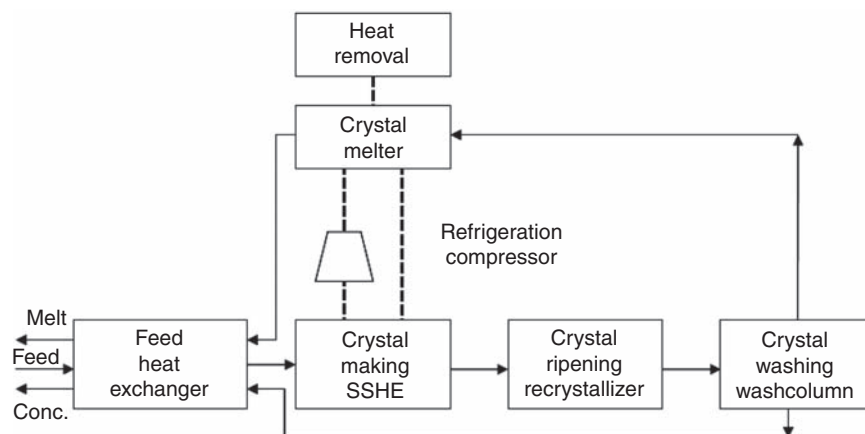


Figure 1. Schematic diagram for a single-stage freeze concentration process. Source: Best and Vasavada (1993).

(at 40 h), 44 wt% for whole milk (at 30 h), 46.5 wt% for whey protein concentrate (at 24 h) and 51 wt% for whey permeate (at 26 h). Skim milk contains the highest level of casein, whey protein concentrate the highest level of whey proteins and permeate the highest level of lactose. High viscosity was reported to be the limiting factor for the freeze concentration of both skim milk and whole milk. The highest concentrations were obtained in those product streams with the highest lactose and whey protein concentrations. This suggests that casein and not whey protein or lactose crystallization is the factor that limits the maximum obtainable concentration for dairy products.

In another work, Hartel and Chung (1993) investigated the critical subcooling required before stable secondary ice nuclei form, as an important design parameter for suspension freeze concentration systems (Table 1). As the whey protein content increased, the critical subcooling temperature also increased, suggesting that whey proteins act as inhibitors of secondary nucleation. On the other hand, it was found that lactose had only a slight effect on the critical subcooling temperature for contact nucleation. The observation that whey proteins affect the rate of growth of ice crystals explains the results obtained by Best and Vasavada (1993) with whey permeate. If the critical subcooling temperature increases, it may also affect the microscopic structure of the crystal surface. For instance, if the growth rate of ice crystals is decreased due to the presence of whey proteins (Shirai et al., 1985), the surface may become smoother.

The Concept of Heat Balance and the Importance of the Optimal Heat Balance Conditions

Hartel and Espinel (1993) concluded from freeze concentration experiments with skim milk that large, easily separated crystals can be grown in short times, if optimal heat balance conditions can be maintained throughout the freeze concentration process. The authors thus claim that a better understanding of ice crystal formation and growth in fluid food products will help to determine the optimal operating conditions for production of large, smooth and spherical ice crystals needed for efficient separation in the shortest possible time. Optimizing the growth of ice crystals in suspension freeze concentrators includes enhancing the rate of ice crystallization, which is done by maintaining high heat-transfer rates in the recrystallizer. Parameters to be controlled are: (i) the refrigerant temperature, which is continually lowered in response to the decreasing bulk temperature to maintain high rates of heat transfer, (ii) crystal growth rates and (iii) the agitation rate, which is continually increased to counteract decreases in heat and mass transfer coefficients during concentration.

A phenomenon that may affect the optimal heat balance conditions during the process of suspension freeze concentration is the ice fouling (build-up of an ice layer on the cooler surface), which is an important factor to be considered in the design of new equipment. Freeze concentration of reconstituted whole milk (10 wt% TS) within a suspension crystallizer led to the problem of ice fouling on the subcooled metal surface, which greatly impeded the heat removal from the bulk solution (Qin et al., 2003). The heat transfer surface is more likely to trigger the first batch of nuclei since it is at a lower temperature than the bulk solution (Huige and Thijssen, 1972; Stocking and King, 1976; Hartel and Chung, 1993). Teflon coating of the cooler surface delayed the occurrence of ice fouling (Qin et al., 2003).

Normally, the systems for suspension freeze concentration use a scraped surface heat exchanger (SSHE) in which the nucleation of ice crystals occurs. In this type of equipment, the ice is removed with blades (scrapers) attached to a central rotating shaft. By scraping the ice off the surface during their rotation, these scrapers maintain a clean surface for heat transfer while also providing high heat transfer rates. However, ice removal is achieved at a very high cost (Habib and Farid, 2007).

In a recent study, Qin et al. (2009) studied and modeled ice nucleation from aqueous solutions on a subcooled solid surface. The most important conclusion for suspension freeze concentration of the modeling studies is that the induction time of ice fouling is correlated with the degree of supercooling at the cooling wall. This correlation can be used to estimate the critical time interval between two scraping actions in the SSHE in order to optimize the process and save energy.

On the other hand, the use of a fluidized bed heat exchanger (FBHE) has been presented as an economically competitive alternative to the SSHE (Habib and Farid, 2006). The FBHE consists of particles fluidized in a vertical column by an upflowing liquid, which by their 'gentle scouring' action prevent the build up of fouling ice layers on the column walls (Figure 2).

A number of stable operating points were obtained for a fluidized bed with particles of 5 mm, at different concentrations of skim milk (10, 13, 14, 15 and 16 wt% TS contents). Ice formed during processing of whole milk provided more resistance to removal than in the case of skim milk. This is probably caused by the increase in fat content (Habib and Farid, 2007).

Most of the ice growth in a multi-stage system is carried out at lower concentrations so that growth rates are much faster than in a single stage system (Zhang and Hartel, 1996). The authors designed a six-stage multilayer freezer for freeze concentration of liquid milk (Figure 3) based on the fact that mixing effects and density differences between ice and liquid concentrate generate a natural counter-current flow, providing suitable conditions for ice crystal growth and ice-liquid separation. Ice crystals are generated

Table 1. Studies of suspension freeze concentration with milk.

Fluid, author and year of publication	Aim of study	Equipment	Results
Skim milk (Hartel and Espinel, 1993)	To determine the conditions for an optimal heat balance (refrigerant temperature, agitation rate and surface area of crystals) at which optimal crystal growth can be attained	Suspension batch crystallizer	The most important parameters for maintaining optimal heat balance conditions are crystal surface area and refrigerant temperature. Large crystals can be grown (600–800 µm diameter) in reasonably short times in batch crystallizers when the heat balance conditions are maintained at high levels
Lactose solutions, whey extract solution with the addition of whey protein (Hartel and Chung, 1993)	To investigate formation of secondary or contact nuclei	Stirred batch crystallizer	Fluid dairy products had different critical subcooling temperatures and this temperature increases as the TS content of each type of product increases. A statistical analysis shows that the whey protein contents of these products play the dominant role in determining the critical subcooling temperature for secondary nucleation
Skim milk (Dickey et al., 1995)	Practical feasibility of direct freeze concentration of milk for plants of various sizes was estimated from pilot plant data	Pilot plant up to 1140 L of skim milk with central vessel of Ø61 cm	Skim milk was concentrated to approximately 20 wt% TS by direct freezing and ice filtration. The concentrate was vacuum evaporated to 45 wt% TS with the same equipment
Skim milk (Zhang and Hartel, 1996)	To evaluate the influence of different heat and mass transfer conditions on the performance of a multilayer freezer for freeze concentration of liquid milk by varying refrigerant temperatures and agitation rate	Six-stage multilayer freezer: chambers mounted as layers in a column with individual refrigerant jackets	– Freeze concentration in the multilayer freezer was feasible for low concentration (10–17 wt%) skim milk Both refrigerant temperature and stirring rate influenced efficiency of ice crystallization, with refrigerant temperature having a larger influence
Reconstituted whole milk (Qin et al., 2003)	To study factors influencing the ice fouling on a subcooled stainless steel surface	Stirred suspension crystallizer	Ice fouling will eventually take place after ice particles are introduced in the crystallizer The fouling induction time is mainly determined by the degree of wall supercooling and the mass of ice seeds
Pasteurized whole milk (Park et al., 2006)	Freeze concentration of whole milk through carefully controlled ice recrystallization	Multi-stage freeze concentrator in stainless steel vessel (Ø135 × 200 mm)	Maximum TS obtained: 32.7 wt%, ripening time of 8 h in a second stage process. Milk solid yield decreased according to the solute concentration
Skim and whole milk (Habib and Farid, 2007)	Freeze concentration of milk in a FBHE	FBHE	Milk can be freeze concentrated with a FBHE using 5 × 5 mm ² particles for ice removal in the process. This was possible at a bed porosity of 0.78 in which skim milk of 13 wt% TS content was concentrated to more than double its original TS content (27 wt% TS)

mostly in the bottom stage, float up as they grow and are removed as slurry from the top stage. Skim milk is fed to the top stage, which flows down as it becomes more concentrated and is discharged from the bottom stage. The main objective was to control the ice crystal growth and heat removal; hence, the equipment was based on controlling heat and mass transfer rates. The crystallizer was designed to maintain rapid crystal

growth by controlling the temperature (driving force) along the length of the column. The major factors influencing heat and mass transfer were the refrigerant temperature, the concentration of skim milk, the stirring rate and the rates of feed, discharge and ice removal. However, mass transfer was not the limiting factor over the experimental range of this study, primarily due to the relatively low solute concentrations (about

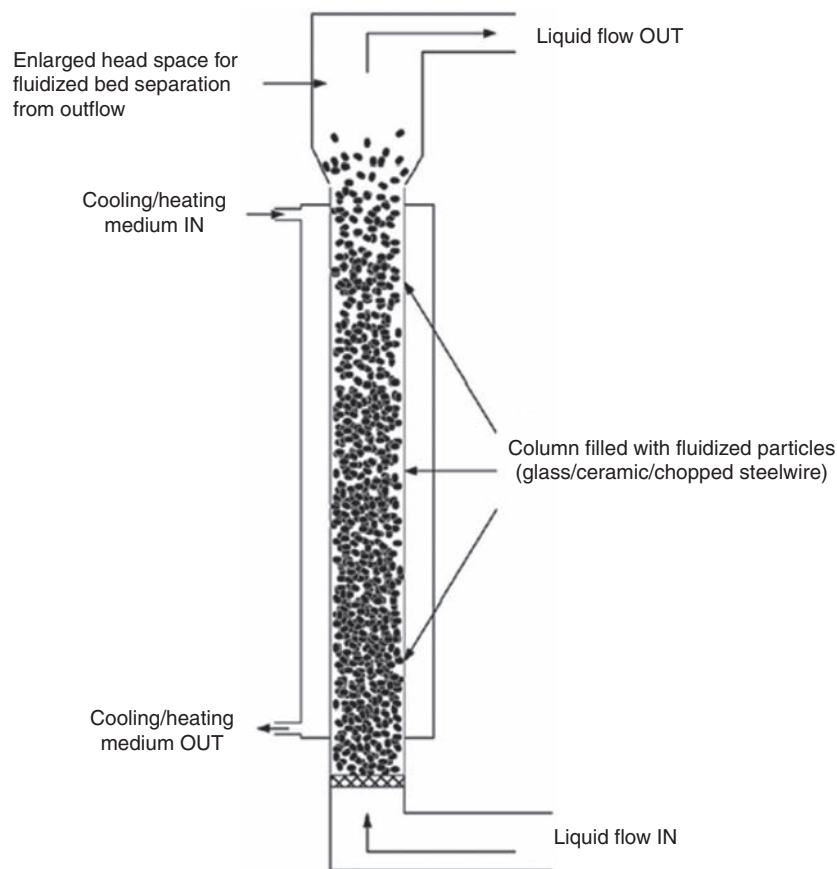


Figure 2. A single tube FBHE. Source: Habib and Farid (2006).

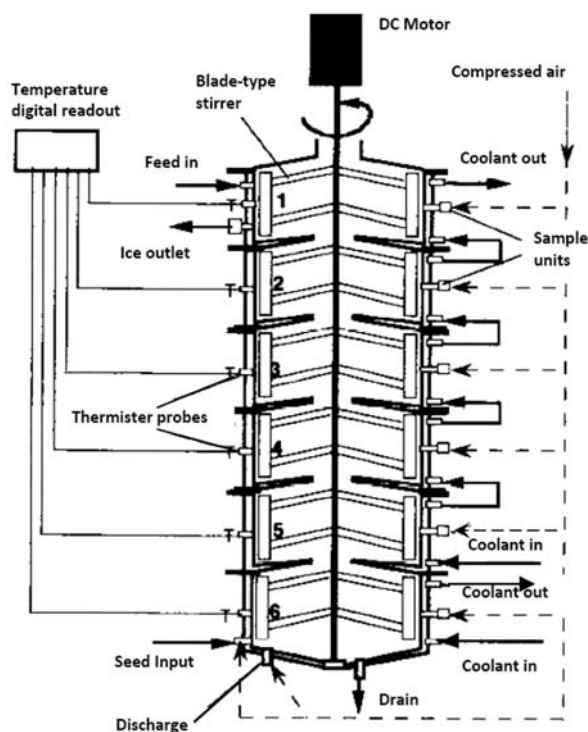


Figure 3. Six-stage multilayer freezer for freeze concentration of liquid milk. Source: Zhang and Hartel (1996).

10 wt% for feed and 17 wt% for discharge) and viscosities. Fresh skim milk experienced the highest refrigerant temperature and the most concentrated product in stages 5 and 6.

Studies on separation and concentration of dairy products that may be of interest for a thorough understanding of the technology of suspension freeze concentration are described by Dickey et al. (1995) in Table 1.

FREEZE CONCENTRATION BY BLOCK AND LAYER CRYSTALLIZATION

Aider et al. (2007) studied the freeze concentration of whole cheese whey (lactoserum). The method used was a series of successive concentrations (Table 2). In the freeze concentration of cheese whey, the maximum TS content that could be reached was about 35 wt%. Moreover, after the fourth level of the freeze concentration process, an abrupt increase of the total protein content was observed to an average value of 19.87 wt%. For the interpretation of this experiment, it is important to mention that whey proteins are able to bind a great number of water molecules. This is possible mainly via hydrogen bonds. By increasing the

Table 2. Studies of freeze concentration by layer and block crystallization with milk.

Fluid, author and year of publication	Aim of study	Equipment	Results
Milk, milk fat (Peters-Erjawetz et al., 1999)	To show that solid-layer melt crystallization has the potential to fractionate milk fat	Dynamic mode: falling film crystallizer (cooled tube) Static mode: jacketed beaker, cooled tube and magnetic stirrer	The solid-layer crystallization process can produce milk fat fractions with melting profiles similar to those produced commercially by the suspension process
Fresh whole milk and reconstituted whole milk (Chen et al., 1999)	To investigate the distribution of solute inclusion in ice (spatial uniformity) considering velocity over the plate, ice growth rate and refrigerant temperature	Crystallizer: refrigerant channel covered by a 1 mm thick stainless steel plate	Fresh whole milk gives similar solute inclusions in ice at different locations as the reconstituted whole milk Solute inclusion and ice growth rate increase along the solution flow direction The ice growth rate increases with increasing temperature difference between the entering solution and the plate
Whole and skim milk (Chen and Chen, 2000)	To correlate as a simple generalized empirical equation, the relationships among average solute distribution coefficient, average ice growth rate, solution velocity, bulk concentration of solute, and effective molecular weight	Crystallizer: refrigerant channel covered by a 1 mm thick stainless steel plate	The average ice growth rate, the solution velocity, the bulk solute/solid concentration and the effective molecular weight are the four key factors in determining average distribution coefficient With an $r^2 = 0.91$ a simple generalized empirical equation was found
Fresh whole milk whey (Aider et al., 2008)	To concentrate milk whey by freeze concentration technology and to compare between gravitational and microwave-assisted thawing of the frozen solution	Container cooled in a freezer at -20 ± 2 °C by indirect cooling. four freeze concentration levels	Whey concentrate with $37.72 \pm 0.69\%$ of total dry matter was obtained Proteins were concentrated up to $6.49 \pm 0.31\%$ (w/v) Microwave-assisted thawing was an efficient procedure and was faster than gravitational thawing
Skim milk whey (Aider et al., 2009a)	To study a freeze concentration procedure of skim milk whey and to compare the results with those obtained using whole milk whey	Container cooled in a freezer by indirect contact (cold air recirculation). Four freeze concentration levels	Total dry matter in the concentrated fraction: $38.54 \pm 0.47\%$ (w/v) Total protein content in the concentrated fraction increased linearly At the fourth freeze concentration stage, lactose content in the ice increased drastically and reached a mean value of $14.19 \pm 0.89\%$
Skim acid milk whey (Aider M. et al., 2009b)	To optimize the whey freeze concentration process by minimizing the amount of dry matter entrapped in the ice fraction and to study the emulsifying and foaming properties of the concentrated whey as function of the freeze concentration cycle	Container cooled in a freezer at -18 ± 1 °C by indirect cooling	Acid whey with an initial total dry matter content of $5.71 \pm 0.01\%$ (w/w) was freeze concentrated up to $24.68 \pm 0.03\%$ in three freeze concentration cycles. Emulsion stability index (ESI) increased by increasing freeze concentration cycle from 19.82 ± 0.58 min up to 72.49 ± 16.37 min Foaming capacity of the freeze concentrated whey decreases by increasing freeze concentration cycle, whereas the foaming stability increases
Whole cheese whey (lactoserum) (Aider et al., 2007)	To develop a new method for whey concentration and to study the impact on the physicochemical properties of the final product	Container cooled in a freezer by indirect contact (cold air recirculation). Five freeze concentration levels	Maximum solids content that could be reached was about 35% (w/w) Total protein content in the ice decreased linearly until the freeze concentration level no. 4 Lactose concentration increased in the ice fraction at the end of the freeze concentration (15.37% w/w) Nutritional, biological and protein qualities of the product were preserved
Pasteurized whole milk (Kramer et al., 1971)	To study the migration of solids as influenced by position of a sample cell in relation to the cooling medium (migration of solids in ascending and descending freezing)	A square sample cell made from cellophane film	For whole milk, no changes in TS were observed in ascending and descending freezing

concentration of proteins in the solution, the interstitial water becomes less available for freezing. During the separation procedure of the ice phase and the concentrated aqueous solution (defrosting period), the frozen phase remains rich in proteins. This phenomenon decreases the effectiveness of the process, but it is possible to optimize it by choosing the optimal quantity of solution to be defrosted. Lactose was not accumulated in the concentrated fraction, and the major part of lactose was retained in the ice fraction.

In order to compare the results with those obtained using whole milk whey (Aider et al., 2009a), skim milk whey was freeze concentrated using a similar protocol as that used in Aider et al. (2007) in which lactose and lipids were more concentrated in the ice fraction than in the concentrated fraction, and this high lactose concentration in the ice fraction was correlated with the high concentration of lipids in the same fraction (Table 2). However, in the study with skim milk whey, this phenomenon was not observed. It thus seems that the fat fraction is a key parameter that influences the lactose distribution between the two fractions (ice and concentrate). During the four freeze concentration stages, the ratio of the total protein to total dry matter in the concentrated fraction and in the ice fraction were very similar.

Even though microwave-assisted thawing was an effective treatment for total dry matter and protein concentration, it showed a negative effect by inducing aggregate formation and protein denaturation, especially of β -lactoglobulin. It is important to notice that the concentrated fraction was rich in potassium ions.

Other studies on separation and concentration for dairy products that may be of interest for a thorough understanding of the technology of layer freeze concentration are described by Peters-Erjawetz et al. (1999).

The Solute Inclusion in Ice

The spatial distribution of solute inclusion in ice formed from falling flows on a sub-cooled surface (stainless steel plate) was investigated by Chen et al. (1999) (Table 2). In this study, fresh whole milk and reconstituted whole milk with solid concentrations of up to 25 wt% were freeze concentrated and the effects of solution velocity over the plate, ice growth rate and refrigerant temperature were considered.

The average spatial distribution was defined by the average distribution coefficient (\bar{K}):

$$\bar{K} = \frac{W_{ice}}{W_s} \quad (1)$$

where W_{ice} is the solute mass fraction in ice (wt%) and W_s the solute mass fraction in the bulk solution (wt%). Fresh whole milk gives similar solute inclusions in ice at

different locations as the reconstituted whole milk at the same concentration with an ice growth rate of 0.1–0.4 $\mu\text{m/s}$. The solute entered in the ice was 6.2 ± 8.3 wt% from 25.1 wt% whole milk, 3.3 ± 5.7 wt% from 20.5 wt% whole milk and about 0.4 ± 1.8 wt% from 13.6 wt% fresh and reconstituted whole milk at a low ice growth rate. In freeze concentration of whole and skim milk in a crystallizer comparable to that used by Chen et al. (1999), the average distribution coefficient \bar{K} (equation 1) was correlated (Chen and Chen, 2000) with the average ice growth rate, solution velocity, bulk concentration of solute and effective molecular weight as a generalized equation (2):

$$\bar{K} = a + b\text{FPD} + c\text{FPD}^2 + d \frac{\bar{V}_{ice}}{us.\infty^{0.5}} \quad (2)$$

where $a = -0.10$, $b = 0.32$, $c = -0.04$, $d = 0.12$, FPD is the freezing point depression ($^{\circ}\text{C}$), \bar{V}_{ice} the average ice growth rate (m/s) and $us.\infty$ the fluid velocity (bulk solution) (m/s). It is interesting to notice that for all the solutions tested, the effects of the solute/solid concentration and effective molecular weight (kg/kmol) on \bar{K} can be represented by the effect of freezing point depression ($^{\circ}\text{C}$).

Kramer et al. (1971) studied the migration of solids as influenced by the position of the sample to be cooled in relation to the cooling medium (Table 2). The cooling medium was placed above (descending freezing) and below (ascending freezing) the sample. In descending freezing, the soluble solids migrate downward depending on the rates of ice formation and gravity. Pasteurized whole milk (12 wt% TS) was used to represent a fat-protein emulsion system and no changes were observed in TS in ascending and descending freezing (distance from cooling surface: <1.27 cm = 12.1 wt% and 3.81–5.08 cm = 11.5 wt%). It was found that, when the freezing occurs very fast, the solutes present in the milk may get captured within the ice crystals and this may be the reason why no change was found in solute concentration.

CONCLUSION AND PERSPECTIVES

The main investigations on freeze concentration of dairy products have focused on the optimal heat balance conditions in order to obtain large and easily separated crystals in a short time and on the effect of the casein and whey proteins on the rate of freeze concentration. Lactose and lipids are more concentrated in the ice fraction than in the concentrated fraction and the casein seems to be the factor that limits the rate at which milk can be freeze concentrated. In the case of milk whey, the increase in whey protein concentration decreases the ice growth rate.

By suspension freeze concentration, skim milk has been concentrated up to 40 wt% TS and whole milk up to 44 wt% TS. By block freeze concentration, cheese whey and whey proteins have been concentrated up to 35 wt% TS and up to 6.49% (w/v), respectively.

High viscosity is a limiting factor for the freeze concentration of both skim milk and whole milk. It is also of importance that nutritional, biological and protein qualities of the product are preserved.

Process efficiency can be improved by:

- Ice recycling to extract the maximum amount of solids.
- Removing high molecular weight solutes (by ultrafiltration) that inhibit ice crystallization (for example casein and whey protein).
- Optimizing heat transfer and scraping speeds in SSHEs or using FBHEs.
- Using ice separation methods combined with reverse osmosis to reduce solute recovery costs, developing a method to recycle partially melted discharged crystals.

It is noticed that less studies have been published on dairy products than on fruit juices; this is probably caused by the fact that freeze concentration of dairy products is more difficult. The presence of fats in the form of an emulsion is a main challenge in the operation of freeze concentration equipment. For example, butter-fat separation in the wash column interferes with proper wash-column operation during concentration of whole milk.

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REFERENCES

- Aider M., de Halleux D. and Akbache A. (2007). Whey cryoconcentration and impact on its composition. *Journal of Food Engineering* **82**: 92–102.
- Aider M., de Halleux D. and Melnikova I. (2008). Gravitational and microwave-assisted thawing during milk whey cryoconcentration. *Journal of Food Engineering* **88**: 373–380.
- Aider M., de Halleux D. and Melnikova I. (2009a). Skim milk whey cryoconcentration and impact on the composition of the concentrated and ice fractions. *Food and Bioprocess Technology* **2**: 80–88.
- Aider M., de Halleux D. and Melnikova I. (2009b). Skim acidic milk whey cryoconcentration and assessment of its functional properties: impact of processing conditions. *Innovative Food Science and Emerging Technologies* **10**: 334–341.
- Best D.E. and Vasavada K.C. (1993). Freeze concentration of dairy products phase 2. Final report. Dairy Research Foundation, Elk Grove Village, IL.
- Chen P. and Chen X.D. (2000). A generalized correlation of solute inclusion in ice formed from aqueous solutions and food liquids on sub-cooled surface. *The Canadian Journal of Chemical Engineering* **78**: 312–318.
- Chen P., Chen X.D. and Free K.W. (1999). An experimental study on the spatial uniformity of solute inclusion in ice formed from falling film flows on a sub-cooled surface. *Journal of Food Engineering* **39**: 101–105.
- De Boer R. and Hiddink J. (1980). Membrane processes in the dairy industry. *Desalination* **35**: 169–192.
- Dickey L.C., Craig J.C., Radewonuk E.R., McAloon A.J. and Holsinger V.H. (1995). Low temperature concentration of skim milk by direct freezing and vacuum evaporation. *Journal of Dairy Science* **78**: 1369–1376.
- Flesland O. (1995). Freeze concentration by layer crystallization. *Drying Technology* **13**: 1713–1739.
- Habib B. and Farid M. (2006). Heat transfer and operating conditions for freeze concentration in a liquid–solid fluidized bed heat exchanger. *Chemical Engineering and Processing* **45**: 698–710.
- Habib B. and Farid M. (2007). Freeze concentration of milk and saline solutions in a liquid–solid fluidized bed Part I. Experimental. *Chemical Engineering and Processing* **46**: 1400–1411.
- Habib B. and Farid M. (2008). Freeze concentration of milk and saline solutions in a liquid–solid fluidized bed Part II. Modelling of ice removal. *Chemical Engineering and Processing* **47**: 539–547.
- Hartel R.W. and Chung M.S. (1993). Contact nucleation of ice in fluid dairy products. *Journal of Food Engineering* **18**: 281–296.
- Hartel R. and Espinel L.A. (1993). Freeze concentration of skim milk. *Journal of Food Engineering* **20**: 101–120.
- Heldman R.D. (2003). *Encyclopedia of Agricultural, Food and Biological Engineering*. New York: Marcel Dekker Inc, pp. 385–387.
- Hernández E., Raventós M., Auleda J.M. and Ibarz A. (2009). Concentration of apple and pear juices in a multi-plate freeze concentrator. *Innovative Food Science and Emerging Technologies* **10**: 348–355.
- Huige N.J.J. and Thijssen H.A.C. (1972). Production of large crystals by continuous ripening in a stirred tank. *Journal of Crystal Growth* **13**: 483–487.
- Kramer A., Wani K., Sullivan J.H. and Shomer I. (1971). Freeze concentration by directional cooling. *Journal of Food Science* **36**: 320–322.
- Müller M. and Sekoulov I. (1992). Waste water reuse by freeze concentration with a falling film reactor. *Water Science and Technology* **26**: 1475–1482.
- Park S.-H., Kim J.-Y., Hong G.-P., Kwak H.-S. and Min S.-G. (2006). Effect of ice recrystallization on freeze concentration of milk solutes in a lab-scale unit. *Food Science Biotechnology* **15**: 196–201.
- Peters-Erjawetza S., Ulrich J., Tiedtke M. and Hartel R.W. (1999). Milk fat fractionation by solid-layer melt crystallization. *Journal of the American Oil Chemists' Society* **76**: 579–584.
- Qin F., Chen X. and Free K. (2009). Freezing on subcooled surfaces, phenomena, modeling and applications. *International Journal of Heat and Mass Transfer* **52**: 1245–1253.
- Qin F., Russel A., Chen X. and Robertson L. (2003). Ice fouling on a subcooled metal surface examined by thermo-response and electrical conductivity. *Journal of Food Engineering* **59**: 421–429.

- Raventós M., Hernández E., Auleda J.M. and Ibarz A. (2007). Concentration of aqueous sugar solutions in a multi-plate cryo-concentrator. *Journal of Food Engineering* **79**: 577–585.
- Sánchez J., Ruiz Y., Auleda J.M., Hernández E. and Raventós M. (2009). Review. Freeze concentration in the fruit juices industry. *Food Science and Technology International* **15**: 303–315.
- Shirai Y., Nakanishi K., Matsuno R. and Kamikubo T. (1985). Effects of polymers on secondary nucleation of ice crystals. *Journal of Food Science* **50**: 401–406.
- Stocking H.J. and King C.J. (1976). Secondary nucleation of ice sugar solutions and fruit juices. *AIChE Journal* **22**: 131–140.
- van Mil P.J.J.M. and Bouman S. (1990). Freeze concentration of dairy products. *Netherland Milk Dairy Journal* **44**: 21–31.
- Zhang Z. and Hartel R.W. (1996). A multilayer freezer for freeze concentration of liquid milk. *Journal of Food Engineering* **29**: 23–28.