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Article

Progressive freeze concentration of skimmed milk in an agitated vessel: Effect of the coolant temperature and stirring rate on process performance

Isabella de Bona Muñoz[1], Ariadna Rubio[2], Mónica Blanco[3], Mercè Raventós[2], Eduard Hernández[2] and Elane Schwinden Prudêncio[1]

Abstract

The aim of this study was to investigate the freeze-concentration of skimmed milk by a progressive freeze concentration process. The progressive freeze concentration procedure was performed at three different temperatures (-5, -10, and $-15\,°C$) and stirring rates (0, 500, and 1000 r/min). The solids concentration was determined and used for calculations of the efficiency of the process, concentrated yield, and experimental results validation. A general linear model was applied to determine the influence of the two factors studied, namely coolant temperature and agitation speed. In all tests, it was possible to concentrated skimmed milk with total solids content higher ($P < 0.05$) than ultra-high temperature skimmed milk. The highest concentration ($P < 0.05$) was achieved at low coolant temperature ($-15\,°C$) and high agitation speed (1000 r/min). The coolant temperature and the stirring rate both had a significant effect ($P < 0.05$) on the results of efficiency of the process and concentrated yield. Nevertheless, the parameter that showed the most significant effect in our study was the stirring rate. The tests presented a good fit since the root mean square values were below 25%. The freezing point temperatures of the concentrated milk fractions were lower than that of skimmed milk. Finally, the best-operating conditions in our study were achieved using a high coolant temperature ($-5\,°C$) and high mechanical stirring (1000 r/min), which was also the variable with the lowest ($P < 0.05$) retention of solids in the ice fraction. In our study, the progressive freeze concentration technique showed promise as an alternative for the dairy industry since it makes the development of new dairy products possible.

INTRODUCTION

The search for new technologies in the food industry is increasing since consumers are continually seeking differentiated products in the market (Ferrão et al., 2016). To meet this demand for new products, concentration techniques are gaining acceptance among processing industries such as food and dairy processing

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(Burdo et al., 2008). Concentration processes can also reduce costs of transportation, handling, and storage since those costs are based on the product's mass (Okawa et al., 2009). Seeing that the milk contains high water content, many processes of milk transformation could be improved if concentration were used (Abd El-Gawad and Ahmed, 2011). Evaporation is the traditional method most often used to concentrate milk, but this operation requires a high amount of energy (2260 kJ/kg) to remove water (Amran et al., 2016). Furthermore, this high processing temperature can diminish the nutritional value and other desirable properties of milk. According to Aider and Ounis (2012), heating milk to a temperature of 70 °C can cause irreversible aggregation of heat sensitive **[AQ1]** proteins. So, one alternative to replace this traditional technology of milk concentration could be a freeze concentration process (Aider et al., 2009), which uses low processing temperatures.

The freezing concentration process involves decreasing the solution temperature below its freezing point, with the purpose of avoiding the eutectic temperature and therefore the solidification of all product components. Unlike traditional methodologies, the energy required during the freezing concentration process is considered relatively low, at ∼335 kJ/kg water (Jusoh et al., 2008). In this process, the latent heat of fusion is lower than the latent heat of vaporization of the evaporation process (Hunter and Hayslet, 2002; Qin et al., 2006). Since there is no high temperature involved, it is possible to preserve the composition of the concentrated liquid food through the freezing concentration process (Aider and de Halleux, 2009; Petzold and Aguilera, 2013). Among freeze concentration processes, progressive freeze concentration (PFC) is one of the most important for liquid foods. The main difference between the PFC process and other systems of freeze concentration is related to ice crystal growth. The PFC system produces ice crystals layer by layer on a cooled surface until a single and substantial crystal block is formed (Jusoh et al., 2009; Miyawaki et al., 2005). Since there is only one single ice crystal block, the separation between the ice crystal and the concentrated solution occurs quickly, resulting in a low operation cost (Petzold and Aguilera, 2013). In a vertical PFC system, the solution is poured into a cylindrical agitated tank equipped with a cooling jacket. The ice layer grows on the cooling wall, and mechanical stirring can be used to reduce the solute occlusion in the ice (Osorio et al., 2018). Thus, the highest performance and efficiency of a freezing concentration process depend strongly on finding the optimum operating conditions.

Ojeda et al. (2017) and Osorio et al. (2018) evaluated the influence of the following operational variables in the freeze concentration process of sucrose and ethanol-water solutions: initial concentration of the solution (C_i), refrigerant temperature (T), and stirring speed (ω). Yahya et al. (2017) recently studied the effect of the coolant temperature and the agitation speed on a PFC system for sugarcane juice. However, all these operational variables, as well as the milk submission to the PFC system, have still not been evaluated. Therefore, our study aimed to evaluate the performance of PFC of skimmed milk in an agitated vessel while varying coolant temperature and stirring rate.

MATERIALS AND METHODS

Skimmed milk

For use in the freezing concentration process, UHT skimmed milk with protein, lipid, and carbohydrates contents equal to 3.1, 0.3, and 4.8 g 100 g^{-1} of skimmed milk was obtained from a local supermarket in the area of Barcelona (Spain). Twenty-seven liters of UHT skimmed milk, stored at room temperature (20 ± 5 °C) until the realization of the experiments, were employed during the experimental performance. Before starting the tests, the milk was cooled to a temperature below 0 °C.

Experimental set-up

The tests were performed with experimental PFC equipment as described by Ojeda et al. (2017) (Figure 1). It is comprised of a jacketed tank (1) with an inner diameter of 115 mm, 230 mm height, and 2400 mL capacity. The tank was isolated using polyurethane foam (2) to limit heat exchange. The refrigerant that circulated the tank comes from a thermostatic bath (3) (Polyscience 9505, USA) which allows for temperature maintenance between −30 °C and 150 °C ± 0.5 °C and which also has a temperature control system (4). The refrigerant used in this work was a 50/50 vol% blend of ethylene glycol and water (5). In the tank (6), the UHT skimmed milk temperature reached

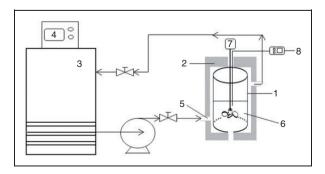**Figure 1.** Experimental set-up of PFC system.

values below $0\,^{\circ}\text{C}$. When appropriate, the milk was stirred using a mechanical stirrer (7) RGL-100 (Heidolph Instruments, Germany) powered by a speed control system PCE-DT62 (PCE Deutschland GmbH, Germany) with 0.05% of precision and $0.1\,\text{r/min}$ of resolution. During the tests, the milk temperature was registered using a digital data logger (8) Testo 925 (TESTO, Germany) and measured with a type K thermocouple with a precision of $\pm 0.1\,^{\circ}\text{C}$ placed into the middle of the tank.

Experimental design for the PFC

Figure 2 shows the experimental plan used in this study. It was proposed to evaluate the influence of different coolant temperatures (-5, -10, and $-15\,^{\circ}\text{C}$) and stirring rate (0, 500, and $1000\,\text{r/min}$) on the process. For this reason, the process was carried out through just the first stage of concentration for each variable analyzed. Each test was performed using $1\,\text{L}$ of skimmed milk and for each variable three repetitions were performed. The duration of the test was $60\,\text{min}$ to ensure proper contact between the jacketed tank and the fluid to be concentrated and also to guarantee appropriate operation of the mechanical stirrer. At the end of each test, the concentrated milk (CM) fractions were weighted using a precision balance KB 1200-2N (KERN, Germany). In the same way as the CM fraction, the ice (I) fraction was also removed and weighted. The formed ice was melted to obtain a representative result for the concentration of solids. Finally, the two fractions (CM and I) were stored in plastic containers and maintained in a freezer until analysis.

Solids concentration

The total solids content of initial skimmed milk, concentrated milk (CM) fractions, and ice fractions (I) were determined according to Floren et al. (2016) with modifications. The solids concentration was first estimated by $^{\circ}\text{Brix}$ using an Atago refractometer (DBX-55, Japan) with an accuracy of 0.1 and measurement range of 0 to $55\,^{\circ}\text{Brix}$ a temperature of $20 \pm 5\,^{\circ}\text{C}$. Previously, a standard curve of total solids content ($g\ 100\,g^{-1}$) against $^{\circ}\text{Brix}$ readings was plotted using different concentrations of skimmed milk. The curve points were constructed from samples consisting of skimmed milk powder with an equal composition to the initial skimmed milk used in the freeze concentration tests through applying different dilutions (2%, 5%, 10%, 15%, 20%, 25%, and 30%). Through a linear regression ($y = 2.2435x - 0.8875$, $R^2 = 0.998$) the $^{\circ}\text{Brix}$ results of the tests were converted and expressed as total solids content ($g\ 100\,g^{-1}$). Finally, the calculations related to the performance of the PFC process were carried out using the total solids data.

Freeze concentration efficiency

The freeze concentration efficiency relates to the increase in the solids concentration of the concentrated fraction relative to the solids content retained in the ice fraction, i.e., the lower the solids concentration present in the ice fraction, the more concentrated the solution will be. The efficiency of each variable studied was calculated by equation (1), as follows

$$\textit{Efficiency (eff) (\%)} = \frac{Cf - Ch}{Cf} \times 100 \qquad (1)$$

where C_f is the total solids content ($100\,g^{-1}$) of the CM fraction, and C_h is the total solids content ($g\ 100\,g^{-1}$) of the ice (I) fraction formed at the end of each test.
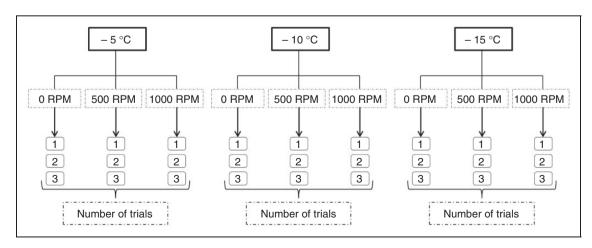

Figure 2. Experimental design for the PFC tests.

Concentrated yield (Y)

The concentrated yield was defined as the ratio of the mass of solute present in the separated liquid to the mass of solute present in the original solution. The concentrated yield of total solids was calculated by Miyawaki et al. (2016) and Moreno et al. (2013), using equation (2)

$$Y(\%) = \frac{C_f \cdot m_f}{C_i \cdot m_i} \times 100 \qquad (2)$$

where C_f is the total solids content (g $100\,g^{-1}$) of the CM fraction, C_i is the initial total solids content (g $100\,g^{-1}$) of the skimmed milk, m_f is the concentrated milk mass (g), and m_i is the initial milk mass (g).

The average ice growth rate

The average ice growth rate ($\mu m \cdot s^{-1}$) was calculated from the mass of the ice sheet at the end of the process, the solids concentration in the ice, the process time, the ice density and the dimensions of the ice area on the vessel, according to equation (3) (Chen et al., 1998).

$$\bar{v}_{ice} = \frac{m_{ice}}{A \cdot t \cdot \rho_{ice}} \cdot (1 - C_h) \cdot 10^6 \qquad (3)$$

where m_{ice} is the mass of ice (kg), ρ_{ice} is the density of pure ice (kg/m^3), C_h is the total solids content (g $100\,g^{-1}$) of the ice, t is time taken for ice growth (s) and A is the area covered by ice within the vessel (m^2).

Experimental results validation

As employed by Belén et al. (2012), Burdo et al. (2008), and Sánchez et al. (2011), a mass balance was calculated in order to validate the experimental results. A comparison between the mass balance and theoretical data was realized by calculation of the predicted ice mass ratio (W_{pred}) (kg of ice per kg of skimmed milk), as defined in equation (4).

$$W_{pred} = \frac{Ci - Cf}{Ch - Cf} \qquad (4)$$

where C_i is the initial total solids content (g $100\,g^{-1}$) of the skimmed milk, C_f is the total solids content (g $100\,g^{-1}$) of the concentrated milk (CM) fraction, and C_h is the total solids content (g $100\,g^{-1}$) of the ice (I) fraction.

The deviation between experimental and theoretical data was expressed as root mean square (RMS) deviation, as described in equation (5).

$$RSM(\%) = \sqrt{\frac{\sum \left(W_{exp} - W_{pred}/W_{exp}\right)^2}{N}} \times 100 \qquad (5)$$

where W_{exp} and W_{pred} correspond to the experimental and the predicted ice mass ratio, respectively, while N is the number of repetitions performed.

Freezing point depression

Aiming to observe the influence of the PFC process in both the UHT skimmed milk and the concentrated milk (CM) fractions, the freezing point curves were determined in triplicate for each sample. The experimental set up used (Figure 3) consisted of three closed tubes (1) with 35 mL of sample (2) placed in a freezer (3) (Fricon Model THC 520, Portugal) at $-20\,°C$. A type K thermocouple (4) TESTO model 177-T4 (TESTO, Germany), with accuracy $0.1\,°C$, previously calibrated with distilled water, was placed in the middle of the tubes to register the temperature changes at intervals of 1 min.

Statistical analysis

All results were expressed as a mean and standard deviation. A general linear model (GLM) was performed to determine the influence of the two studied factors, i.e., temperature (T) and agitation or stirring rate (A), on the results of C_f, *eff*, and Y. Tukey's test was also applied to compare all possible pairs of means. $P < 0.05$ was the threshold used to determine whether results were significantly different. Minitab 18 for Windows (Minitab Inc. State College, PA, USA) was used for the statistical analyses.
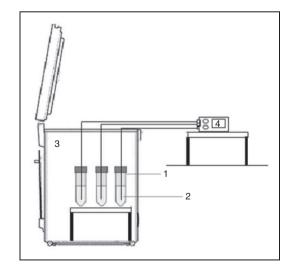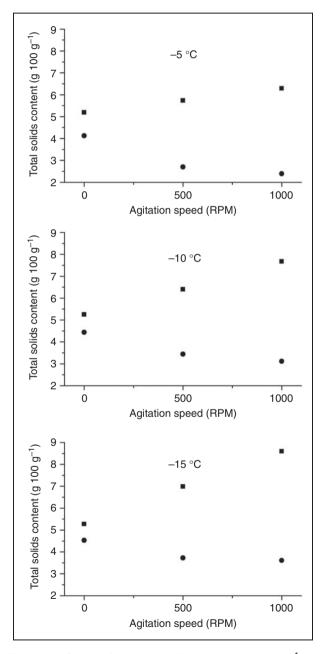

Figure 3. Device to determine the freezing point curves of the UHT skimmed milk and the concentrated milk (CM) fractions.

RESULTS AND DISCUSSION

Figure 4 shows that the total solids contents were higher in the CM fraction than in the I fraction, although in all the tests, the retention of solids was observed in the ice fraction. However, it is noteworthy that this behavior is closely related to the composition and fluid behavior at low temperatures. The initial total solids content of skimmed milk used in the tests was equal to 5.00 ± 0.03 g 100 g^{-1}. In comparison with this
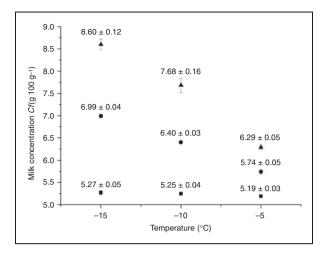

Figure 4. Graph of the total solids content (g 100 g^{-1}) from the concentrated milk (■) and the ice fractions (●) about the agitation speed (0, 500, and 1000 r/min).

value, the highest solids concentration (8.60 ± 0.12 g 100 g^{-1}) in the liquid fraction was achieved in the test performed at $-15\,°C$ and 1000 r/min of stirring ($P < 0.05$). The lowest value (5.19 ± 0.03 g 100 g^{-1}) was observed in the test conducted at $-5\,°C$ without stirring ($P < 0.05$). It is possible that the lower concentration obtained in the test at $-5\,°C$ and without stirring is due to the low ice production and the high retention of solids in the ice fraction (4.13 ± 0.02 g 100 g^{-1}). So, it can be stated that the tests performed with these operating conditions ($-5\,°C$ and 0 r/min) resulted in a negative effect on milk concentration. In addition, it is possible that in the freeze concentration process the solution is concentrated due to the water removal as ice, which is indicated by the higher ice formation observed at the lowest temperature ($-15\,°C$).
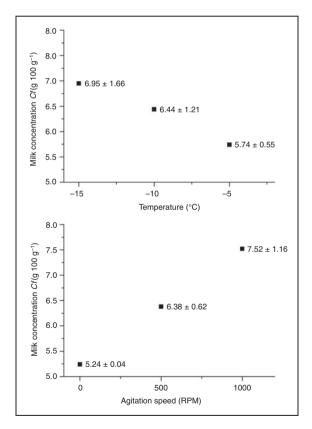
Regarding the concentration of solids in the ice, the best condition had a high coolant temperature ($-5\,°C$) and high agitation (1000 r/min). This result indicates that the concentration of solids in the ice is determined by the advance of the ice front and by the agitation rate. Shirai et al. (1998) stated that reducing the advance of the ice and increasing the stirring rate, the ice concentration decreased. The average ice growth rate of the tests, calculated according to Chen et al. (1998), is within the interval between 1.47 and $4.02\,\mu m\,s^{-1}$. Moreno et al. (2014) and Ojeda et al. (2017) observed similar results during the freeze concentration of coffee and sucrose solutions, respectively. So, these average ice growth values were lower than the critical value of approximately $8\,\mu m\,s^{-1}$ reported by some authors (Moreno et al., 2014; Petzold et al., 2016). These works suggest that at a speed higher than $8\,\mu m\,s^{-1}$, the freezing is too fast to expect a significant separation of the concentrated fraction, and in these conditions, the solids are trapped in the ice during the freezing phase.

With regard to the stirring rate, in all the trials, the concentration of solids in the ice decreased ($P < 0.05$) with the increase of the stirring rate. The tests performed without any stirring showed higher retention ($P < 0.05$) of solids in the ice fraction. According to Amran et al. (2016), higher stirring rates can increase the rate of mass transfer at the interface. This means that the solute will be distributed better in the solution, hence reducing the concentration of solids in the ice fraction.

Through the GLM test, it was possible to analyze the effect of the two factors (T and A) on the response variable C_f (Figure 5). So, it was possible to verify that the temperature was dependent on the agitation speed (0, 500, and 1000 r/min). The solids concentration (Cf) presents a similar behavior at any temperature without any stirring. The results shown in Figure 6 suggest a more significant influence of the agitation speed on the

Figure 5. Graph of the effect of the interaction between temperature (T) and agitation speed (A) on the total solids content (g 100 g^{-1}) at 0 r/min (■), 500 r/min (•), and 1000 r/min (▲).

Figure 6. Graph of the individual effect of the two studied factors (Temperature and Agitation speed) on the total solids content (g 100 g^{-1}).

solids concentration than the coolant temperature (−5, −10, and −15 °C). Ojeda et al. (2017) observed similar behavior by the concentration of sucrose solutions while using the same freeze concentration system employed in the present study.

Table 1 shows the results of freeze concentration efficiency (eff). For all the coolant temperatures studied (−5, −10, and −15 °C), a stirring rate of 1000 r/min had the highest efficiency. Thus, it obtained an ice fraction with lower solids presence and a concentrated fraction with more solids content. In contrast, the lowest freezing concentration efficiency was observed under conditions without stirring.

The GLM test was also applied to study the effect of temperature and agitation factors on eff. In this case, since the homogeneity of variances assumption was not met, Welch's test was conducted and followed by the Games-Howell method for pair wise mean comparisons. Then, as observed by Jusoh et al. (2013), it was noted that only the agitation speed (A) factor was significant for the eff. These results also suggest that there was a significant influence of the stirring rate on the PFC system. This is probably because the mechanical stirring induces convection, improving the water flow from the concentrated fraction towards the ice fraction (mass transfer) and therefore also heat transfer. Liu et al. (1999) stated that the agitation increases the mass transfer rate of the solutes from the ice front to the liquid fraction due to the fluid stirring. Osorio et al. (2018) related this phenomenon to freezing concentrate composed of ethanol and water; the efficiency remained stable with the increase of the stirring rate. Finally, it was noteworthy that the best efficiency results were obtained at a temperature of −5 °C and agitation speed of 1000 r/min, resulting in the purest ice fraction (2.39 ± 0.14 g 100 g^{-1}).

Regarding the concentrated yield (Y) behavior, the GLM analysis shows that the factors temperature (T), agitation speed (A) and their interaction have a significant effect on Y. In this case, the best values obtained were at a temperature of −5 °C with moderate agitation (500 r/min), and the worst results were obtained at a low temperature (−15 °C) and high agitation (1000 r/min). This behavior may be because at low temperature (−15 °C) and high agitation (1000 r/min), the average ice growth rate was the highest (4.0 μm s^{-1}). Similar results were reported by Amran and Jusoh (2016) in a vertical finned crystallizer of glucose solutions. On the other hand, it is interesting to note that the increase in agitation from 500 to 1000 r/min does not improve the results of Y. A possible explanation according to Figure 4 is that this is due to the small differences between the concentration of solutes retained in the ice at 500 and 1000 r/min. This behavior is in agreement with those reported by Gu et al. (2008) and Ojeda et al. (2017). In light of the results of efficiency (eff) and concentrated yield (Y), it seems that the best conditions for the PFC of skimmed milk are high coolant temperatures and high agitation speed.

Table 1. Results of milk concentration, Ice concentration, efficiency concentration and concentrated yield

Temperature (°C)	Rotation (r/min)	Milk concentration (Cf) (g 100 g⁻¹)	Ice concentration (Ch) (g 100 g⁻¹)	Efficiency (eff) (%)	Concentrated yield (Y) (%)
−5	0	5.19 ± 0.03F	4.13 ± 0.02AB	20.54 ± 0.77D	76.72 ± 1.41B
	500	5.74 ± 0.05E	2.70 ± 0.14EF	53.00 ± 2.19B	83.68 ± 1.56A
	1000	6.29 ± 0.05D	2.39 ± 0.14F	62.07 ± 1.99A	82.26 ± 2.45AB
−10	0	5.25 ± 0.04F	4.44 ± 0.41A	10.65 ± 0.54E	54.83 ± 1.36DE
	500	6.40 ± 0.03D	3.44 ± 0.11D	46.21 ± 1.96C	63.94 ± 3.35C
	1000	7.68 ± 0.16B	3.11 ± 0.21CDE	57.52 ± 0.72AB	57.42 ± 1.71D
−15	0	5.27 ± 0.05F	4.53 ± 0.33A	10.18 ± 1.07E	42.53 ± 0.39F
	500	6.99 ± 0.04C	3.61 ± 0.15BCD	48.44 ± 1.91C	51.43 ± 1.97E
	1000	8.60 ± 0.03A	3.72 ± 0.03BC	56.70 ± 0.58B	37.62 ± 2.25F

Within a column, different uppercase letters denote significant differences ($P < 0.05$) between Cf, Ch, eff, and Y.

Table 2. Experimental results validation for two different factors (temperature and agitation speed)

Temperature (°C)	Agitation speed (r/min)	W_{pred}	W_{exp}	RSM (%)
−5	0	0.20 ± 0.01	0.24 ± 0.01	13.50
	500	0.24 ± 0.03	0.26 ± 0.01	9.02
	1000	0.32 ± 0.02	0.34 ± 0.01	5.98
−10	0	0.35 ± 0.16	0.47 ± 0.01	6.70
	500	0.46 ± 0.01	0.48 ± 0.03	4.77
	1000	0.60 ± 0.02	0.61 ± 0.02	4.38
−15	0	0.54 ± 0.01	0.55 ± 0.07	6.84
	500	0.61 ± 0.02	0.60 ± 0.01	3.10
	1000	0.75 ± 0.01	0.74 ± 0.02	2.66

RMS: root mean square; W_{pred} and W_{exp}: predicted and experimental ice mass ratio (kg if ice per kg of skim milk).

Table 2 shows the experimental results validation evaluated by the comparison of the theoretical (W_{pred}) results with the experimental (W_{exp}), which showed good agreement. The RMS deviation values ranged from 2.66% to 13.50%. By Lewicki (2000), when these values are lower than 25%, they show an acceptable fit. Petzold et al. (2015), Belén et al. (2012), Hernández et al. (2010), and Sánchez et al. (2010) also observed similar results to those in the present study.

The freezing point depression of the UHT skimmed milk, with 5.00 ± 0.03 (g 100 g⁻¹) of total solids content was equal to −0.35 °C. Chen et al. (1996) and Radewonuk et al. (1983) obtained similar results upon analyzing the skimmed milk freezing point at similar solute concentrations. According to Hernández et al. (2009), the freezing point of a liquid is essential and dependent on the concentration and types of solutes present in the solution, i.e., the more concentrated solutions generally have a lower freezing point. This information is critical to design a freeze concentration system since the refrigeration requirements and operating conditions depend to a large extent by the freezing temperature.

In comparison to the freezing point of the UHT skimmed milk (−0.35 °C), all the concentrated milk (CM) fractions showed a decline in freezing point temperature. It is necessarily highlighted, that in the test performed at −15 °C and with a stirring rate of 1000 r/min, the freezing point reached the lowest value (−1 °C), which was also the condition with the highest concentration of solids (8.60 ± 0.12 g 100 g⁻¹).

In a typical cooling curve, the lowest temperature indicates the beginning of nucleation, i.e., the formation of ice crystals. Following that, the temperature increases due to the latent heat of the phase change. Finally, the highest temperature reached after that temperature increase is the freezing point of the sample, associated with the growth stage of ice crystals (Auleda et al., 2011). It is clear from this graph (Figure 7) that the high concentration of solids affected
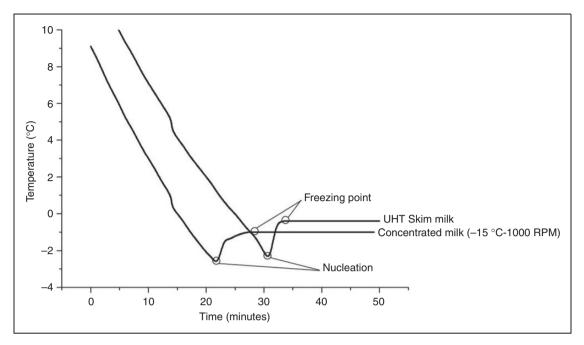
Figure 7. Freezing curves of the UHT skimmed milk and the concentrated milk obtained at −15°C and 1000 r/min.

the freezing point. The same behavior was observed in the freeze concentration of whey (Sánchez et al., 2011), aqueous sugar solutions (Raventós et al., 2007), and fruit juices (Auleda et al., 2011). Through measurement of the freezing point, it is possible to determine the best operating conditions for the PFC since this variable has a direct influence on the efficiency of the process. Therefore, when the freezing point of liquid food is known, it is possible to optimize the cooling capacity of the PFC system.

CONCLUSION

This work has proven that the PFC process is an efficient method to concentrate skimmed milk. The coolant temperature and stirring rate were indicated to have a significant in on the efficiency (e*ff*) and the concentrated yield (*Y*). The best operating conditions in our study were achieved using a coolant temperature of −5 °C and mechanical stirring of 1000 r/min. Overall, the parameter that showed the most significant effect in our study was the stirring rate. Finally, the opportunity to concentrate skimmed milk using an environmentally friendly technology is an attractive opportunity for the dairy industry from an economic, technological, and nutritional perspective.

DECLARATION OF CONFLICTING INTERESTS

The author(s) declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

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