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Dear Editor,

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Optimization of goat milk vacuum-assisted block freeze concentration using response surface methodology and NaCl addition influence

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Abstract

Response Surface Methodology was applied to optimize the effects of freezing time, vacuum conditions, and time under vacuum regarding concentrated yield response, resulting from optimal parameters of the milk vacuum-assisted block freeze concentration process. Additionally, it was verified the NaCl influence, using different salt contents (0.5, 1, 1.5, and 2 %) addition and freezing time of 1 day, vacuum equal to 10 kPa, and time under vacuum 60 min, in goat milk vacuum-assisted freeze concentration performance. The concentrate with 1.5 and 2 % of NaCl addition showed the highest values for the total solids (35.06 and 36.21 g 100 g^{-1}) and protein contents (10.43 and 10.70 g 100 g^{-1}), while the concentrate without NaCl addition concentrated more lactose content (17.42 g 100 g^{-1}). The samples with 1.5 and 2% of NaCl addition reached parameters of the process more satisfactory with a concentrate yield of 85.79 and 92.14 %, concentration percentage of 28 and 32 %, and efficiency of process approximately of 90 %. Finally, the best performance was observed when used 1.5 and 2 % NaCl addition in the goat milk submitted to the vacuum-assisted freeze concentration process.

Keywords: Concentration, caprine milk, optimization, sodium chloride, vacuum thawed.

1. Introduction

Verruck, Dantas, and Prudencio (2019) highlighted that goat milk has attracted huge amounts of attention in the dairy industry and by the researchers in the elaboration because it can be considered a reliable alternative/if not a replacement to cow milk. The increased rates of cow protein allergies of children, credited to the αs1-casein (Albenzio et al., 2012), has encouraged goat product development, such as goat's milk yogurt (Beltrán, Morari-Pirlog, Quintanilla, Escriche, & Molina, 2018) and probiotic fermented goat milk beverages (Mituniewicz-Małek, Zielińska, & Ziarno, 2019). However, the scarcity of scientific information on new technologies concentration use, such as the freeze concentration process, and their effects on composition effects is still evident for goat milk.

The freeze concentration process involves a controlled decrease in temperature of the liquid food below the freezing point, with the purpose to avoid the eutectic temperature where the components of the product frozen (Raventós, Hernández, Auleda, & Ibarz, 2007). The block freeze concentration is one type of freeze concentration processes able to result in a concentrated and an ice fraction separated, which can be separated by the use of external forces, such as the vacuum (Aider & Halleux, 2008). Petzold, Orellana, Moreno, Cerda, and Parra (2016) mentioned that the suction by the use of vacuum as an assisted technique in freezing concentration focuses on improving concentration performance. However, according to the author's knowledge, the goat milk vacuum-assisted freeze concentration has not been pursued before in literature, including the addition of salts in this milk as a step before the freeze concentration process.

The addition of NaCl, in milk in the production of dairy products has a preservative effect, extending shelf life. On the other hand, the NaCl addition into the milk prior to the preparation of a dairy product results, for example, in greater salt homogeneity in the matrix and in a reduction in the salting step during cheese making (Yanachkina, McCarthy, Guinee, & Wilkinson, 2016). It knows the presence of sodium chloride changed the mechanism of freezing and thawing in milk solution by lowering their freezing point. The ice crystal grows in the form of dendritic instead of a planar form. As the ice crystal grew, both sodium chloride and other solutes were concentrated. These concentrated salt solutes were released through the channel formed during the melting of these dendritic ice crystals. However, according to Yee, Wakisaka, Shirai, and Hassan (2004), the concentration index varies according to the amount of sodium chloride added in the milk, which could or not increase the recovered solutes of milk.

Whilst the results of this approach highlight the acceptability of this process for goat milk, they also suggest a potential alternative to the current concentration methods adopted by goat dairy industries. This process, due to its cheaper capital and operating costs, could be an attractive alternative for dairy industries to pursue. Bearing this in mind, firstly was investigated the optimal operating parameters of the goat milk vacuum-assisted freeze concentration process by Response Surface Methodology. In the sequence, the best parameters, founded previously, were used to evaluate the NaCl addition influence about goat milk vacuum-assisted freeze concentration process performance.

2. Material and methods

2.1. Material

Semi-skimmed UHT goat milk (COVAP®, Córdoba, Spain), used as the start material, was obtained from a local supermarket in the area of Barcelona (Spain). The goat milk composition was 9.93 ± 0.01 g total solids 100 g^{-1}, 3.53 ± 0.07 g total protein 100 g^{-1}, 5.08 ± 0.22 g lactose 100 g^{-1} and 1.60 ± 0.03 g fat 100 g^{-1}. All reagents were of analytical grade.

2.2. Goat milk vacuum-assisted block freeze concentration performance

The goat milk (45 mL) placed in plastic tubes was frozen in a static freezer at - 20 ± 1°C. During the freezing process, the external surface of the plastic tubes was covered with thermal insulation made of foamed polystyrene for that the heat transfer mainly occurred unidirectional form. After the freezing process, vacuum goat milk was performed according to the procedure described by Petzold, Niranjan, and Aguilera (2013), to achieve the separation of the most concentrated ice solution. The suction was generated by connecting a vacuum pump to the bottom of the frozen sample at ambient temperature (Fig. 1).

2.2.1. Experimental design

The response surface methodology (RSM) was used to determine the optimum condition for goat milk vacuum-assisted block freeze concentration**.** It was used a central composite design (CCD) with the following three independent factors: vacuum (V) (10, 40 and 70 kPa), time under vacuum (T) (20, 40 and 60 min), and freezing time (F) (1, 7 and 14 days). The variation of independent factors values was obtained by preliminary tests. Based on Park and Drake (2016) and Sharma, Patel, and Patel (2016) were done a preliminary test with this pressure value equal to 74.5 kPa and between 14.6–8.0 kPa, respectively. However, the separation of the concentrated from the ice fraction was observed when the vacuum pump reached 70 kPa until 10 kPa. After these steps, new tests were realized to decide the time under vacuum variation. Therefore, in the pressure equal to 70 kPa the separation of both fractions (concentrated and ice) was only noted after the time under pressure of 20 min. On the other hand, at a pressure equal to 10 kPa with a time under vacuum above 60 min was not possible to continue the vacuum-assisted freeze concentration process due to cracks formation in the ice structure, resulting in absence of vacuum in the freeze concentration process. For this reason, the variation of the independent factor for the times under vacuum choice ranged from 20 to 60 min. It is also pointed out that for economic reasons; the small and medium goat dairy industry stored the goat milk for a minimum of 1 and a maximum of 14 days. Because of this was used freezing time range from 1 to 14 days. For better understanding, were also evaluated the average of these three independent factors, such as vacuum, time under vacuum and freezing time of 40 kPa, 40 min and 7 days, respectively.

The experimental design was composed of seventeen combinations of the independent variables (-1 and 1); eight factorials; six axial; and three repetitions in the central point, as shown in Table 1. All tests are performed in triplicate. In order to avoid systematic errors, all the experiments were carried out at random in order to minimize the effect of unexplained variability on the responses obtained. The response variable was the concentrate yield (Y) using total solids contents. After assessing the fit of the initial regression model, the number of variables was reduced according to stepwise methods. Stepwise selection is an algorithmic procedure used to simplify the initial model and to find a reduced model that best explains the data. The Central Composite Design (CCD) for the two-level and three-factor scheme used is described in Table 1.

The optimal condition was chosen by higher concentrate yield (Y). It is important to note that the pressures indicated in this study (10, 40 and 70 kPa) are absolute pressures (the absolute atmospheric pressure is 101 kPa) and corresponding approximately to 90, 60 and 30 kPa of vacuum.

2.2.2. Influence of NaCl content

Optimal conditions previously determined, such as vacuum, time under vacuum, and freezing time, were employed to evaluate the influence of NaCl in the *g*oat milk vacuum-assisted block freeze concentration performance. Based on the results obtained by Yee, Wakisaka, Shirai, and Hassan (2004), different NaCl content (0.5 g $100g^{-1}$, 1 g $100g^{-1}$, 1.5 g $100g^{-1}$, and 2 g $100g^{-1}$) were added to initial goat milk, which was frozen, and submitted in triplicate to the freeze concentration procedure. In this procedure, the goat milk without NaCl additions was used as a control sample. In this step were obtained from the goat milk with 0 (control), 0.5, 1, 1.5, and 2 g of NaCl addition per 100 g of milk, their concentrate and ice fractions. Therefore, the concentrate and the ice fractions were denoted as follows: concentrate control and ice control; concentrate 0.5 and ice 0.5; concentrate 1 and ice 1; concentrate 1.5 and ice 1.5; concentrate 2 and ice 2, respectively. The total solids, protein, and lactose contents were determined for initial goat milk, and for all concentrate and ice fractions.

2.3 Physicochemical analysis

The total solids content was estimated by °Brix using an Atago refractometer (DBX-55, Japan) with an accuracy of 0.1 and measurement range of 0 to 55 °Brix a temperature of 20 ± 5 °C, according to Muñoz et al. (2018) and Floren, Sischo, Crudo, and Moore (2016), with some modifications. Firstly, a standard curve of total solids content (g 100 g^{-1}) against °Brix readings was plotted using different concentrations of semi-skimmed goat milk. The curve points were constructed from samples consisting of freeze-dried semi-skimmed goat milk by applying different dilutions (5%, 10%, 15%, 20%, 25%, 30%, 35%, 40%, and 50%). Through a linear regression (y = 0.9285x + 0.2764, R^2 = 0.999) the °Brix results of the tests were converted and expressed as total solids content (g 100 g^{-1}).

Protein contents (g 100 g^{-1}) were carried out by the Kjeldahl method, converting the sample nitrogen content to protein content by a factor equal to 6.38 (AOAC, 2005). The lactose content procedure was realized according to Schuster-Wolff-Bühring, Michael, and Hinrichs (2010), with modifications. Hewlett Packard 1100 Series HPLC System (Agilent Technologies, Waldbronn, Germany) with tracer carbohydrate (250 × 4.6 mm, 5 μm) *column* (Teknokroma, Sant Cugat del Vallès, Barcelona, Spain) and C-8 column and refraction index as detector was used for determination. The mobile phase was a mixture of acetonitrile (Panreac Química SLU, Castellar del Vallès, Spain) and distilled water (75:25). The flow rate and column temperature were maintained as 1.3 mL min^{-1} and 28 °C, respectively. Before the determinations, a portion of 1 mL samples was diluted with 8 mL of distilled water and mixed. Thus, 0.5 mL of Carrez Reagent 1 and 2 were added and mixed for 1 min. The mixture was allowed to settle for 15 min, and subsequently, filtered by a nylon syringe filter (0.45 μm of diameter pore) (Agilent, Santa Clara, California, United States). Each sample was prepared and injected in triplicate.

2.4. Concentrate yield (Y)

Goat milk vacuum-assisted block freeze concentration performance and influence of NaCl content were evaluated by the concentrate yields (Y), which were calculated in accordance with Miyawaki, Liu, Shirai, Sakashita, and Kagitani, (2005), and Moreno, Hernández, Raventós, Robles, and Ruiz (2013), using the Equation 1.

$$Y\ (\%) = \left(\frac{concentrate\ fraction\ total\ solids\ (\mathrm{g\ 100\ g^{-1}})\ x\ concentrate\ fraction\ mass\ (g)}{initial\ goat\ milk\ \ total\ solids\ (\mathrm{g\ 100\ g^{-1}})\ x\ initial\ goat\ milk\ mass\ (g)}\right)\ x\ 100 \qquad (1)$$

2.5. Concentration percentage (CP) and efficiency of process (*eff*)

In order to elucidate the influence of different NaCl contents about goat milk, the concentration percentage (CP) and the efficiency of the process (*eff*) were calculated using Equation 2 and 3, respectively.

$$CP\ (\%) = \left(\frac{initial\ mass\ of\ frozen\ fraction\ (g) - final\ mass\ of\ ice\ fraction\ (g)}{initial\ mass\ of\ frozen\ fraction\ (g)}\right)\ x\ 100 \qquad (2)$$

$$eff\ (\%) = \left(\frac{concentrate\ fraction\ total\ solids\ (\mathrm{g\ 100\ g^{-1}}) - ice\ fraction\ total\ solids\ (\mathrm{g\ 100\ g^{-1}})}{concentrate\ fraction\ total\ solids\ (\mathrm{g\ 100\ g^{-1}})}\right)\ x\ 100 \qquad (3)$$

2.6. Results validation

According to Belén, Sánchez, Hernández, Auleda, and Raventós, (2012), Burdo, Kovalenko, and Kharenko, (2008), and Sánchez, Hernández, Auleda, and Raventós (2011), the experimental results were validated by the experimental mass balance of each sample calculation. The experimental results were compared with the theoretical value from NaCl content influence, using Equation 4, where W_{pred} is the predicted ice fraction mass ratio (kg ice/kg goat milk). To determine the deviation between experimental and theoretical data was calculated the root mean square deviation (RMS) (Equation 5), where W_{exp} and W_{pred} are the ratios of experimental and predicted ice mass, respectively, and *N* is the number of test repetitions.

$$w_{pred} = \frac{initial\ goat\ milk\ tatal\ solids\ (\mathrm{g\ 100\ g^{-1}}) - \ concentrate\ fraction\ total\ solids\ (\mathrm{g\ 100\ g^{-1}})}{ice\ fraction\ total\ solids\ (\mathrm{g\ 100\ g^{-1}}) - concentrate\ fraction\ total\ solids\ (\mathrm{g\ 100\ g^{-1}})} \quad (4)$$

$$RMS\ (\%) = 100\sqrt{\frac{\sum\left(\frac{w_{exp}-w_{pred}}{w_{exp}}\right)^2}{N}} \quad (5)$$

2.7. Statistical analysis

The regression coefficients for linear, quadratic, and interaction terms were determined by using multiple linear regressions. The significance of each regression coefficient was judged statistically by computing the t-value from pure error obtained from the replicates at the central point of this experiment. The regression coefficients were then used to generate response surfaces. Results were expressed as a mean ± standard deviation. To determine significant differences ($P < 0.05$) between results of NaCl content influence, it was used one-way analysis of variance (ANOVA), and Tukey studentized range test. All statistical analyses were performed using Minitab 18 for Windows (Minitab Inc. State College, PA, USA).

3. Results and discussion

3.1. Experimental design

The responses obtained for concentrate yield (Y) from the seventeen experiments are shown in Table 1. The *P*-values of the reduced model are shown in Table 2, which shows that all the individual effects in the reduced model were significant ($P < 0.05$). Regarding the quadratic terms, the time under vacuum had an effect ($P < 0.05$). It was also possible to observe that an interaction between the vacuum and freezing time ($P < 0.05$), and between time under vacuum and freezing time ($P < 0.05$) had on effect on the concentrate yield (Y).

The reduced model was obtained in order to eliminate the redundant information by means of the method of variable selection step-by-step (α to enter 0.15, α to remove 0.15). The regression equation of the reduced model is presented in Equation 6, being its R^2 value equal to 0.99. In this equation V is the vacuum (kPa), T is the time under vacuum (min), and F is the freezing time (days).

$$Y = 40.66 - 0.1024\ V - 2.579\ T + 0.601\ F + 0.05415\ T \times T - 0.00774\ V \times F\ 0.01361\ T \times F \quad (6)$$

Fig. 2 (a,b,c) and 3 (a,b) show the contour and surface plot, respectively, elaborated from the regression model, which represents the trend of factor selection for better concentrate yield (Y). These contour and surface plots showed that there was an increase for Y value when time under vacuum was equal to 60 min (Fig. 2c and 3a,b). In Fig. 2 c also was noted that the best concentrate yield was determined when used a vacuum and freezing time equal to 10 kPa and 1 day, respectively, reaching values higher than 77.5 %. A close result for Y (between 76 to 83%) was obtained by Muñoz et al. (2018), after the progressive freeze concentration of skimmed cow milk with an agitated vessel. Tribst et al. (2020) verified that the goat milk freezing time was affected by the milk particle size distribution. According to these authors, the interaction/adsorption of casein micelles with fat globules is responsible for the higher volume of larger particles, indicating that part of the fat globules was clumped or part of proteins were aggregated. Therefore, these clumped/aggregated can compromise the separation of total solids between concentrate and ice fractions. Park, Kim, Hong, Kwak, and Min (2006), evaluating the effect of ice recrystallization on freeze concentration of milk solutes, highlighted that the ice morphology changed during a long freezing time, affecting the solute recovery. These authors affirmed that ice crystal size increased with the freezing time, because most of the ice crystals exhibited an agglomerated and compacted form, reducing the dendritic form crystal, which is founded in shorter freezing times. Therefore, the compacted form may have caused a decrease in the ice channels, reducing the total solids of milk output from the ice fraction. This behavior leads us to believe that the crystal geometry obtained in a long freezing time, is not adequate for the scape of concentrate solution from the ice fraction, resulting in a decrease of the concentrate yield.

3.2. Influence of NaCl content

Under optimal conditions (vacuum equal to 10 kPa, time under vacuum of 60 min, and freezing time of 1 day), the vacuum-assisted block freeze concentration was applied in the goat milk samples without (control) and with different NaCl contents additions. The total solids, protein and lactose contents determined in the concentrate (control, 0.5, 1, 1.5, and 2) and ice fractions (control, 0.5, 1, 1.5, and 2) are shown in Table 3. All concentrates fractions showed higher total solids content than the initial goat milk. However, in relation to the total solids and protein contents, the best freeze concentration performance was observed when added 1.5 and 2%, and 1 to 2 % of NaCl, respectively. These concentrates showed approximately 4 times more ($P < 0.05$) for total solids content and 3 times more ($P < 0.05$) for protein contents, than the initial goat milk. However, in the present study, all total solids contents of concentrates were higher than those determined by Muñoz et al. (2018) and Balde and Aider (2016), for skimmed cow milk, using the progressive freeze concentration and the block freeze concentration, respectively. This behavior is expected because, in accordance with Petzold et al. (2013), the high separation of solids and protein contents obtained by vacuum-assisted freeze concentration is a consequence of using an external driving force (vacuum) that improves the natural separation of gravitational thawing. Between the concentrate fractions, the lower ($P < 0.05$) total solids content was found for the concentrate 0.5. As expected, all ice fractions showed lower ($P < 0.05$) total solids content than goat milk.

Overall, our results indicated in the concentrates fractions that the increase of salt addition resulted in an increase of total solids and protein contents. Yee et al. (2003) stated that the sodium chloride addition, a monovalent salt, influenced the mechanism of freezing and thawing by lowering the freezing point of a protein solution. In this case, this behavior made us believe that the greatest concentration of sodium chloride transition changes the form of ice crystal from planar to dendritic. According to Yee et al. (2003) is expected the growth of dendritic ice crystal during the freezing of solutions with sodium chloride addition, as well as the freezing point becomes lower. Therefore, these dendritic ice crystals melted upon thawing, to form channels that allow the concentrate to be drained out, resulting in the increase of total solids and protein contents.

The lactose content was higher (P < 0.05) for the concentrate control, without NaCl addition, when compared with the others concentrates from goat milk with different NaCl additions. Bhargava and Jelen (1996) concluded that salt addition decreases the lactose solubility. Allan, Gruch, and Mauer (2020) related that the form in which lactose crystallizes into ice crystal is dependent on the water activity, temperature conditions during crystallization, among other factors. Chandrapala, Wijayasinghi, and Vasiljevic (2016) also observed that salts may change the solubility of lactose which leads to supersaturation, thereby affecting the growth of lactose ice crystal. Thus, this fact could have affected the output of lactose from the ice fraction.

The concentrate yield (Y) from the total solids contents is shown in Fig. 4. Concentrate yield highest values ($P < 0.05$) were obtained when used 1.5 % (85.79 %) and 2 % (92.14 %) of NaCl. Similar values were founded using vacuum-assisted block freeze concentration for wine by Petzold et al. (2016) and, for blueberry juice by Orellana-Palma, Petzold, Pierre, and Pensaben (2017b). Similar behavior was also observed for the concentration percentage (CP) values, whose concentrate 1.5 and 2 showed the highest values, is equal to 28 % and 32 %, respectively (Fig. 5). As cited before, these facts are related to the higher content of total solids present in concentrates (Table 3).

The efficiency of the process (*eff*) had a progressive increase ($P < 0.05$) with the increase of the NaCl content (Fig. 6). However, the best *eff* was noted for the process with the control and goat milk with 1.5 and 2 % of NaCl content, which achieved values approximately 90 %. Similar values were reached for the freeze concentration of whey by Aider, Halleux, and Melnikova (2009), and for the skim milk by Balde and Aider

(2016) and Canella et al. (2019). These studies credited the highest *eff* from freeze concentration fractions to their dependence on the total solids contents. The vacuum improved the efficiency over atmospheric conditions in freeze concentration due to the positive effect of pressure difference on the movement of the concentrated liquid fraction in block freeze-concentration, showing conform Pardo and Sánchez (2015) higher efficiency than those in similar processing conditions that used gravity as the separation method.

To validate the experimental results, the mass balance was calculated and compared with the theoretical value from NaCl content influence. The ice mass ratio had an expected downward trend with NaCl addition (Fig. 7), which can be attributed to the NaCl addition. Besides, an agreement was observed between the experimental (Wexp) and predicted (Wpred) ice mass ratios over the NaCl content. With the root mean square (RMS) values were observed a good adjustment of the process since these values were equal to 4.14%, 5.71%, 7.84%, 9%, and 10.52% for the vacuum-assisted block freeze concentration, goat milk without and with 0.5 %, 1%, 1.5 % and 2% of NaCl addition. Lewicki (2000) highlighted that a freeze concentration process is considered an acceptable fit when RMS value was lower than 25 %. Comparing with tests using vacuum-assisted freeze concentration process, Petzold et al. (2013); Petzold et al. (2016); Orellana-Palma, Petzold, Torres, and Aguilera (2017a); and Orellana-Palma et al. (2017b) achieved RMS values of 4.9 % for sucrose solutions; 6.8 % and 9.5 % for wine; 5.1 % and 8.7 % for orange juice; and 3.1 % and 9.6 % for blueberry juice, respectively. Comparing these literature results with our study, we confirm that the goat milk could be submitted to the vacuum-assisted block freeze concentration, considered a recent innovation of food concentration. The results of this approach highlight the acceptability of this process for goat milk is a potential alternative to the current concentration methods adopted by dairy industries. The vacuum-assisted freeze concentration process, due to its cheaper capital, operating costs, and energy consumed, in comparison with the traditional concentration process, such as the vacuum evaporation, is an attractive alternative for goat dairy industries. Balde and Aider (2017) emphasized that the use of freeze concentration is energetically highly interesting, because of the low water latent heat of freezing in comparison with the water latent heat of vaporization (80 kcal/kg *versus* 540 kcal/kg). Moreover, the concentration of goat milk frozen is also important, because of the seasonality of milk production, of the low animal productivity and of the short periods of lactation. Therefore, frozen goat milk is commonly used to overcome these limitations, allowing its storage for days, reaching a compatible volume with dairy production, mainly when the objective is the use of one concentration process. Goat milk concentration shows advantages in terms of processing, packaging, transportation, and handling. Since most changes occur in an aqueous environment, the removal of some part of goat milk water results in milk preservation. It is noteworthy that dairy industries are concerned principally with food preservation and the production of high-quality products.

The results about the influence of NaCl content on the goat milk freeze concentration performance encourage us to recommend the use of concentrates 1.5 and 2 as an ingredient in dairy products development. Therefore, the vacuum-assisted freeze concentration process associated with NaCl addition could have a detrimental effect on the physical and chemical properties of skimmed goat milk, as well as consumer acceptance, which could affect the commercialization potential of these new products.

Sun and Zheng (2006) noted that the flavor and taste of the food products had been substantially increased, after the use of unitary operations which used low temperatures associated with the vacuum. Therefore, it is expected that skimmed goat milk submitted to the vacuum-assisted freeze concentration process could have different sensorial properties. Ranadheera et al. (2019) cited that, for the monitoring and adjustment of sensory characteristics to optimize the acceptability of goat milk products, descriptive tests present great applicability, such as descriptive analysis. This analysis is recognized as an adequate technique to determine the sensory profile of processed foods, thus providing detailed, robust, and reproducible results (Esmerino et al., 2017). In addition, to information on the sensory characteristics of the product, methods that take into account the needs, beliefs, feelings and motivations of consumers are also important for the elaboration of a food product. According to Gambaro (2018), the projective techniques lies in the fact that they lead consumers to express themselves beyond the rational, and allow access to underlying or deep attitudes and emotions, revealing non-conscious or not openly accepted motivations in their buying behavior. These methods do not require training, have a low financial impact, optimize time and resources in dairy industries, and provide information highly correlated with traditional methods (Varela & Ares, 2012), providing a total assessment of products and take all sensory traits into account (Esmerino et al., 2017).

4. Conclusion

Applying the Response Surface Methodology to optimize and evaluate the effects of freezing time, vacuum, and time under vacuum to a frozen goat milk sample it was noted that all factors presented effect in the concentrate yield of the sample. To obtain the higher value of concentrate yield the optimal conditions of vacuum-assisted freeze concentration process are vacuum, time under vacuum, and freezing time equal to 10 kPa, 60 min, and 1 day, respectively. The concentrates fractions from goat milk with 1.5 % and 2 % of NaCl addition are recommended because they showed the best characteristics in relation to total solids and protein contents, which increased 4 and 3 times, respectively, when compared with initial goat milk. The recommendation of both concentrates is also based on their best results obtained to concentrate yield (> 85 %), concentration percentage (≥ 28 %), and efficiency of the process (approx. 90 %) values, as well as a good adjustment of the process, resulting in RMS values less than 11 %.

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Declaration of competing interest

The authors declare that they have no conflict of interest.

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Fig. 1. Vacuum suction procedure.

Fig. 2. Contour plot of the concentrated yield (Y) at 20 (a), 40 (b), and 60 (c) minutes of time under vacuum.

Fig. 3. Surface plot of the interaction effect of (a) time under vacuum (min) and freezing times (days); (b) time under vacuum (min) and vacuum (kPa) on concentrate yield (Y).

Fig. 4. Concentrate yield (Y) as function of NaCl content added to samples (0%, 0.5%, 1%, 1.5%, and 2%) of semi-skimmed goat milk.

Fig. 5. Concentration percentage (CP) as function of NaCl content added to samples (0%, 0.5%, 1%, 1.5%, and 2%) of semi-skimmed goat milk.

Fig. 6. Efficiency of process (*eff*) as function of NaCl content added to samples (0%, 0.5%, 1%, 1.5%, and 2%) of semi-skimmed goat milk.

Fig. 7. Experimental (■) and predicted (- □ -) ice mass ratios as a function as function of NaCl content added to samples (0%, 0.5%, 1%, 1.5%, and 2%) of semi-skimmed goat milk.

Table 1
Central Composite Design (CCD) for three variables levels, and responses of concentrate yield (%) based on vacuum (kPa), time under vacuum (min), and freezing time (days).

		Variables levels[b]			Response
Assay[a]	Type	Vacuum (kPa)	Time under vacuum (min)	Freezing time (days)	Concentrate yield (%)
1	Factorial	10 (-1)	20(-1)	1(-1)	10.02 ± 2.34
2	Factorial	10(-1)	60(1)	1(-1)	77.97 ± 5.48
3	Factorial	10(-1)	20(-1)	14(1)	12.32 ± 0.37
4	Factorial	10(-1)	60(1)	14(1)	74.13 ± 1.04
5	Factorial	70(1)	20(-1)	1(-1)	3.95 ± 0.44
6	Factorial	70(1)	60(1)	1(-1)	73.35 ± 3.56
7	Factorial	70(1)	20(-1)	14(1)	1.16 ± 0.35
8	Factorial	70(1)	60(1)	14(1)	62.54 ± 5.08
9	Axial	40(0)	20(-1)	7(0)	6.81 ± 1.74
10	Axial	40(0)	60(1)	7(0)	79.70 ± 4.65
11	Axial	40(0)	40(0)	1(-1)	22.74 ± 3.94
12	Axial	40(0)	40(0)	14(1)	21.37 ± 4.27
13	Axial	10(-1)	40(0)	7(0)	27.46 ± 0.96
14	Axial	70(1)	40(0)	7(0)	12.07 ± 0.51
15	Center	40(0)	40(0)	7(0)	19.74 ± 0.75
16	Center	40(0)	40(0)	7(0)	15.35± 1.76
17	Center	40(0)	40(0)	7(0)	14.24 ± 1.79

[a]Experiments were conducted randomly.
[b]Coded levels are within brackets

Table 2
Analysis of variance of the values of concentrated yield of semi-skimmed goat milk vacuum-assisted block freeze concentration.

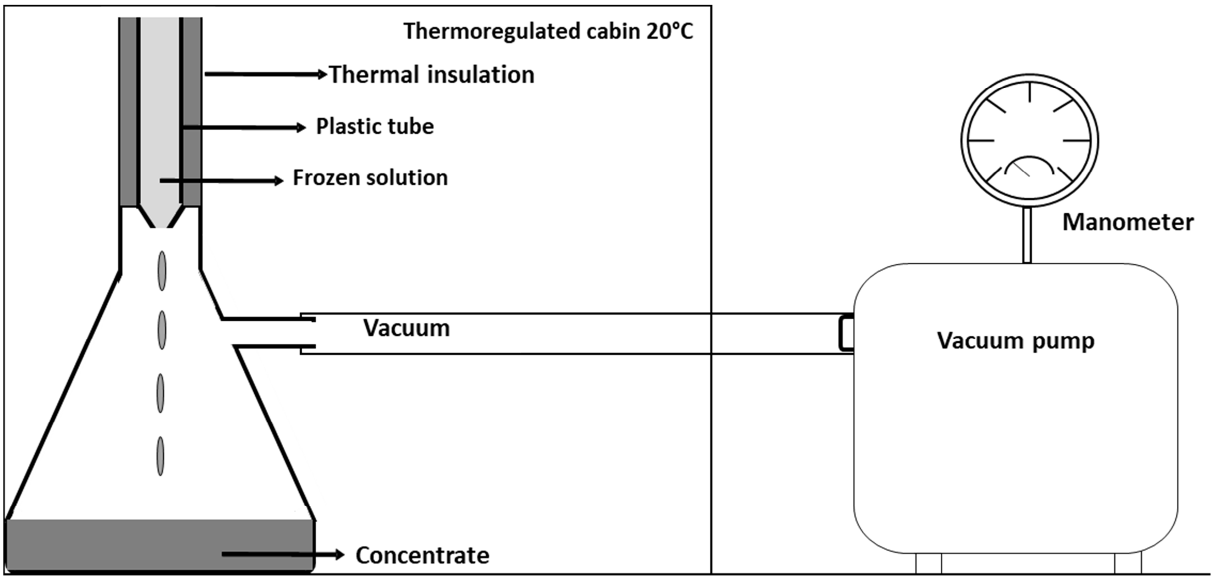
Source	*P* Value
Linear	
Vacuum	0.000*
Time under vacuum	0.000*
Freezing time	0.013*
Quadratic	
Time under vacuum * Time under vacuum	0.000*
Interaction	
Vacuum * Freezing time	0.038*
Time under vacuum * Freezing time	0.016*

*Values significantly different ($P < 0.05$).

Table 3
Total solids, protein and lactose content (g 100 g^{-1}) of initial semi-skimmed goat milk, concentrates, and ice fractions obtained by vacuum-assisted block freeze concentration.

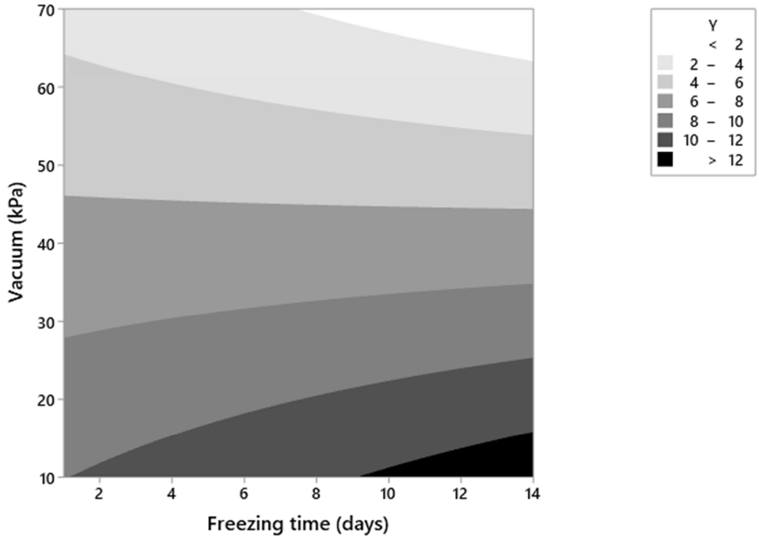
Samples	Total solids (g 100g^{-1})	Protein (g 100g^{-1})	Lactose (g 100g^{-1})
Semi-skimmed goat milk	9.94 ± 0.01^{eA}	3.53 ± 0.07^{cA}	5.08 ± 0.22^{fA}
Concentrate control	32.87 ± 1.31^{bc}	9.43 ± 0.11^{b}	17.42 ± 0.12^{a}
Ice control	3.71 ± 0.53^{F}	1.14 ± 0.08^{D}	1.53 ± 0.08^{D}
Concentrate 0.5	28.07 ± 1.18^{d}	9.31 ± 0.22^{b}	12.45 ± 0.08^{e}
Ice 0.5	9.72 ± 0.04^{B}	3.46 ± 0.16^{A}	2.77 ± 0.09^{B}
Concentrate 1	30.57 ± 1.34^{c}	10.45 ± 0.03^{a}	14.40 ± 0.09^{c}
Ice 1	6.18 ± 1.03^{C}	2.17 ± 0.01^{B}	2.42 ± 0.13^{C}
Concentrate 1.5	35.06 ± 2.76^{ab}	10.70 ± 0.39^{a}	15.63 ± 0.12^{b}
Ice 1.5	5.07 ± 0.20^{D}	1.71 ± 0.04^{C}	1.61 ± 0.14^{D}
Concentrate 2	36.21 ± 1.21^{a}	10.43 ± 0.01^{a}	14.06 ± 0.09^{d}
Ice 2	3.90 ± 0.01^{E}	0.96 ± 0.25^{E}	1.01 ± 0.10^{E}

^{a,b,c} Within a column, means ± standard deviations with different superscript lowercase letters denote significant differences ($P < 0.05$) between the semi-skimmed goat milk and the concentrated fraction of each mixture of milk and NaCl content (g 100 g^{-1}). ^{A,B,C} Within a column, means ± standard deviations with different superscript uppercase letters denote significant differences ($P < 0.05$) between the semi-skimmed goat milk and the ice fraction of each mixture of milk and NaCl content (g 100 g^{-1}). Concentrate control and ice control, Concentrate 0.5, Ice 0.5, Concentrate 1, Ice 1, Concentrate 1.5, Ice 1.5, Concentrate 2, and Ice 2 were the concentrates and ice fractions obtained by vacuum-assited block freeze concentration of semi-skimmed goat milk without and with the addition of 0.5, 1, 1.5, and 2 g of NaCl per 100 g of milk, respectively.

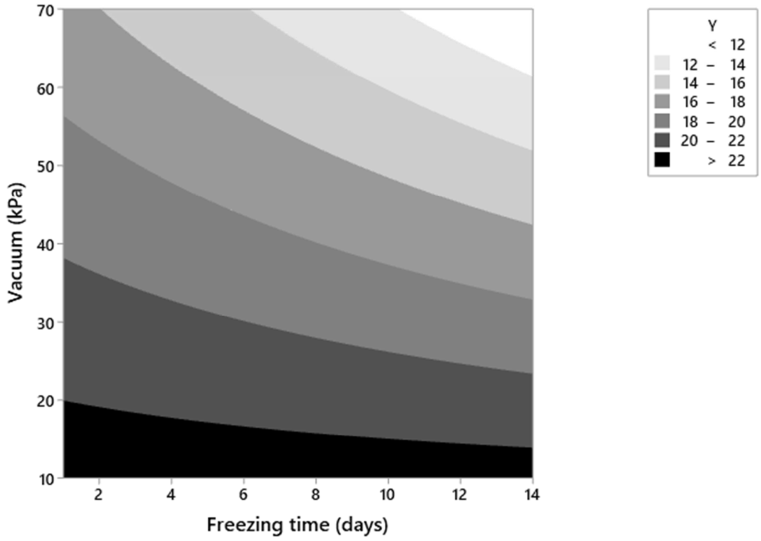


Thermoregulated cabin 20°C
Thermal insulation
Plastic tube
Frozen solution
Manometer
Vacuum
Vacuum pump
Concentrate

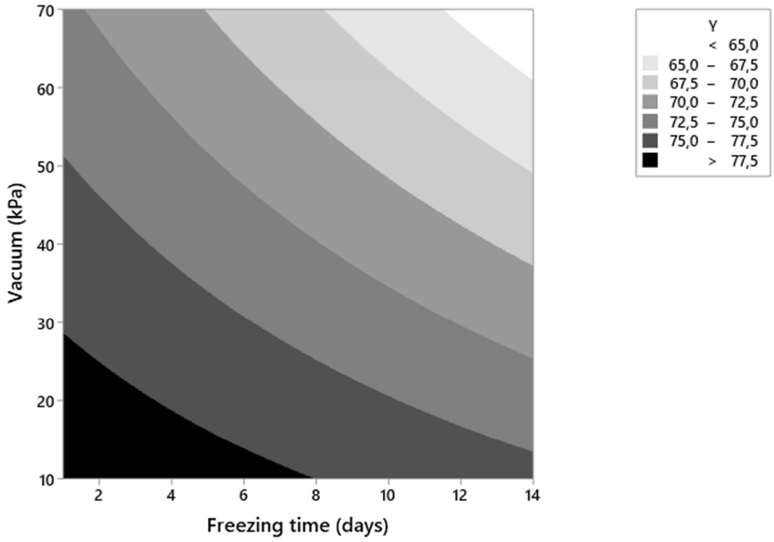
a)


Vacuum (kPa)
Freezing time (days)
Y
< 2
2 – 4
4 – 6
6 – 8
8 – 10
10 – 12
> 12

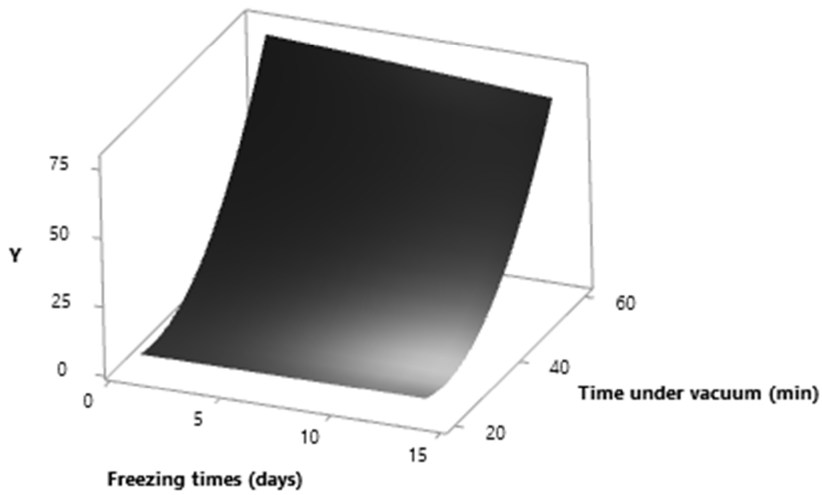
b)


Vacuum (kPa)
Freezing time (days)
Y
< 12
12 – 14
14 – 16
16 – 18
18 – 20
20 – 22
> 22

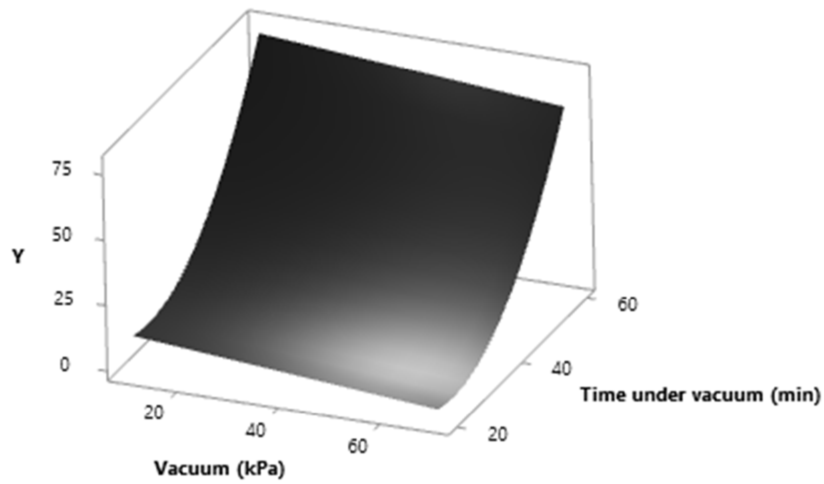
c)

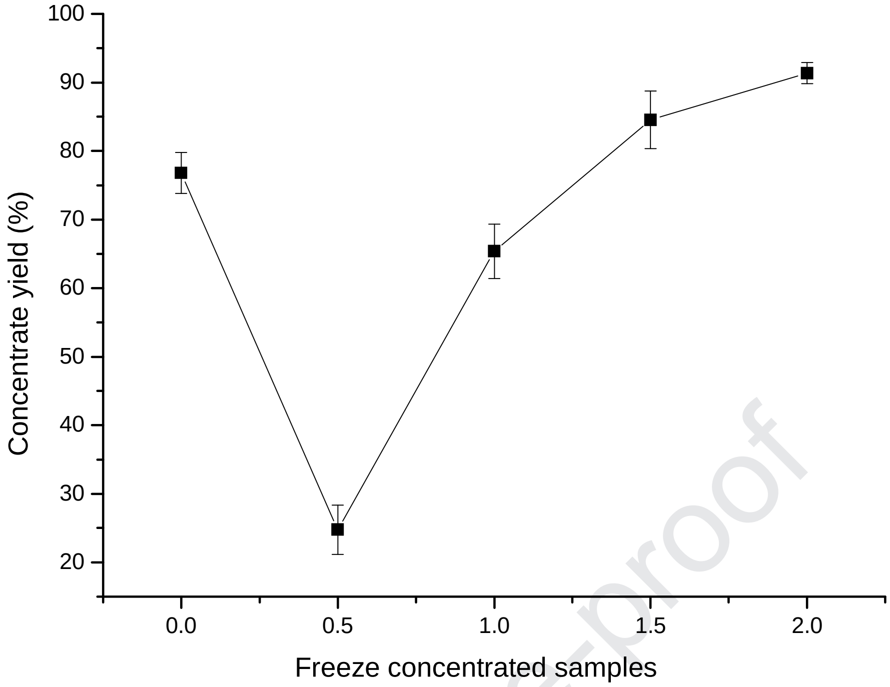

Vacuum (kPa)
Freezing time (days)
Y
< 65,0
65,0 – 67,5
67,5 – 70,0
70,0 – 72,5
72,5 – 75,0
75,0 – 77,5
> 77,5

a)

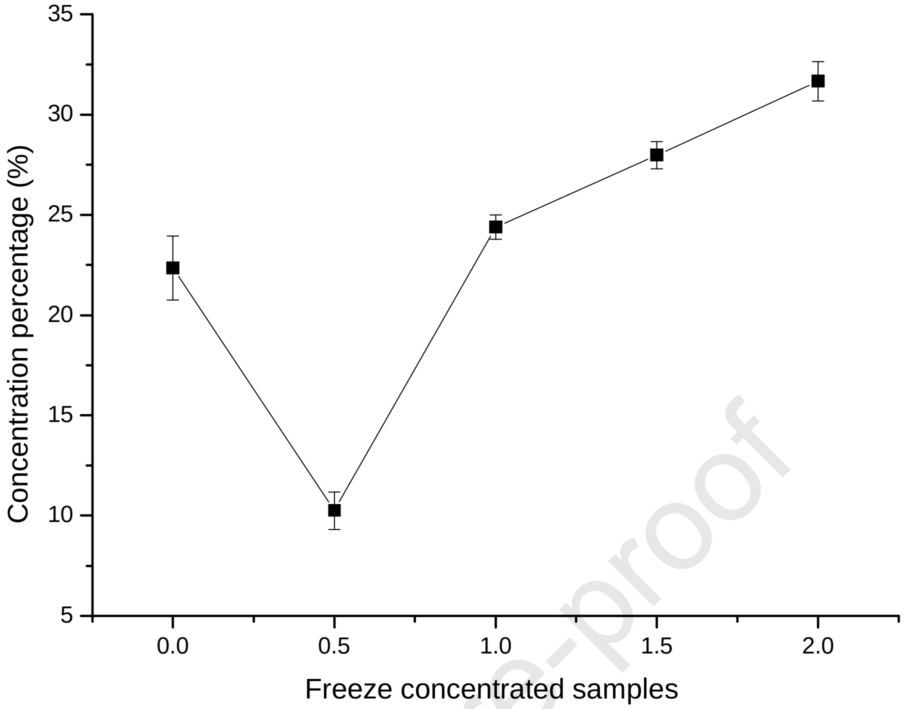

75
50
Y
25
0
0
5
10
15
Freezing times (days)
60
40
20
Time under vacuum (min)

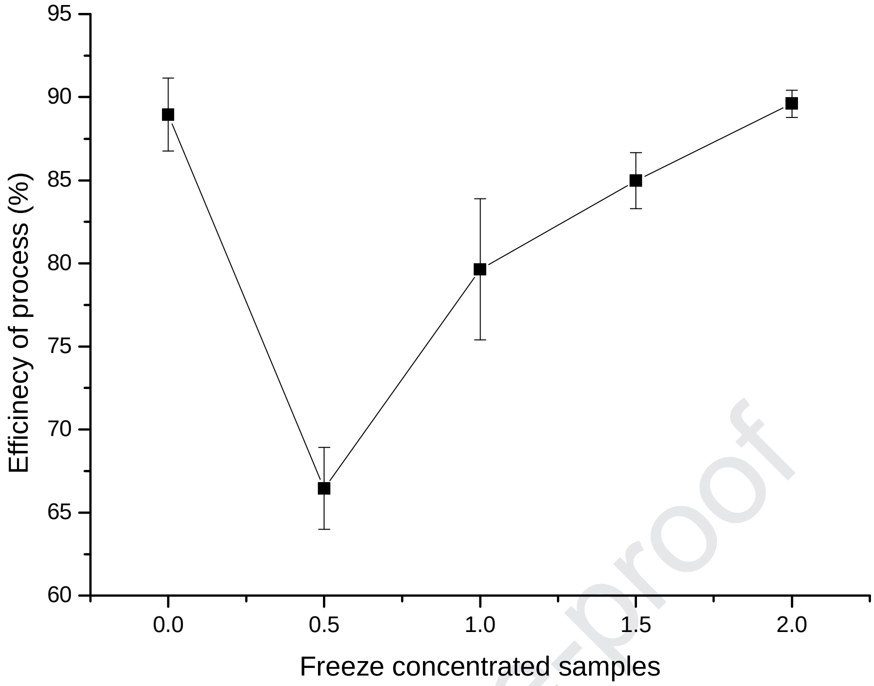
b)

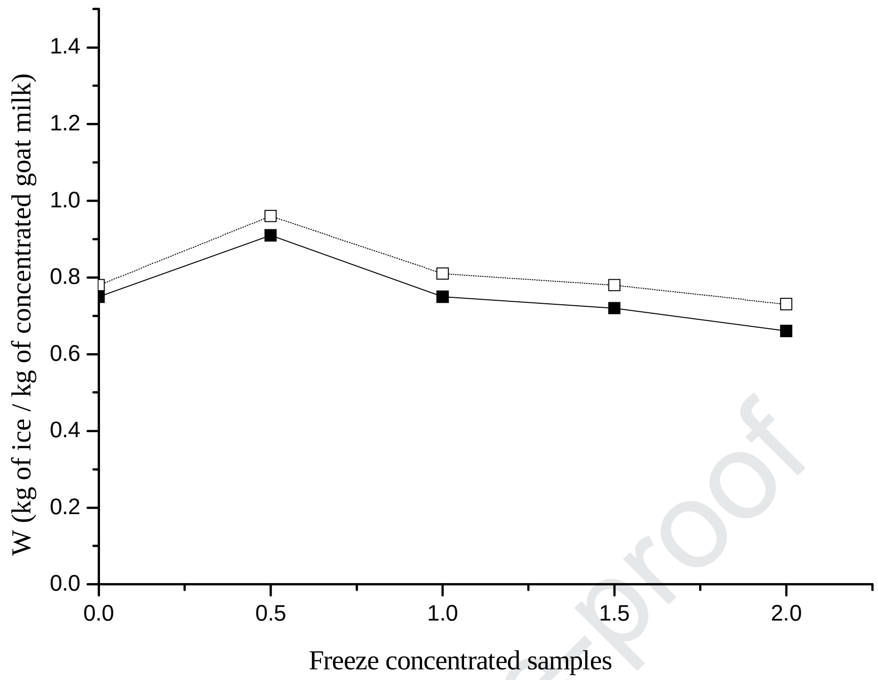

75
50
Y
25
0
20
40
60
Vacuum (kPa)
60
40
20
Time under vacuum (min)



Concentrate yield (%)
100
90
80
70
60
50
40
30
20
0.0
0.5
1.0
1.5
2.0
Freeze concentrated samples


35
30
25
20
15
10
5
Concentration percentage (%)
0.0
0.5
1.0
1.5
2.0
Freeze concentrated samples


95
90
85
80
75
70
65
60
Efficinecy of process (%)
0.0
0.5
1.0
1.5
2.0
Freeze concentrated samples


W (kg of ice / kg of concentrated goat milk)
1.4
1.2
1.0
0.8
0.6
0.4
0.2
0.0
0.0
0.5
1.0
1.5
2.0
Freeze concentrated samples

Vacuum-assisted freeze concentration process influenced about the goat milk concentrated yield.

The optimal condition of goat milk concentration was found for 10 kPa of vacuum, 60 min, and 1 day.

The salt (1.5 and 2 %) into the goat milk increased 4 and 3 times the solids and protein values.

Concentrates from goat milk with 1.5 and 2 % of NaCl were more efficient after concentrations.

Federal University of Santa Catarina

Department of Food Science and Technology

Florianópolis, December 06, 2019.

To: Rakesh K. Singh

Declaration of interest

Dear Editor,

The authors declare that they have no conflict of interest.

Best regards,

Prof. Dr. Elane Schwinden Prudencio

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