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BLOCK FREEZE CONCENTRATION AS A TECHNIQUE AIMING THE GOAT MILK CONCENTRATION: FATE OF PHYSICAL, CHEMICAL, AND RHEOLOGICAL PROPERTIES

Maria Helena Machado Canella*[1], Isabella de Bona Muñoz[1], Eulália Lopes da Silva Barros[2], Callebe Camelo Silva[2], Leandro Antunes de Sá Ploêncio[3], Heitor Daguer[3] & ElaneSchwinden Prudencio[1]*

[1]Department of Food Science and Technology, Agricultural Sciences Center, Federal University of Santa Catarina, Rod. Admar Gonzaga, 1346, Itacorubi, 88034-001, Florianópolis, SC, Brazil.[2]Department of Chemistry and Food Engineering, Federal University of Santa Catarina (UFSC), Technology Center, Florianópolis, Santa Catarina, Brazil
[3]Ministério da Agricultura, Pecuária e Abastecimento, Laboratório Nacional Agropecuário, Seção Laboratorial Avançada de São José (SLAV/SC/LANAGRO/RS), São José, SC 88102-600, Brazil

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ABSTRACT

In the face of the results scarcity for the goat milk processing using emerging and innovative technologies, the results obtained in the present study are relevant, and may in the future be extremely important for goat milk industry. Therefore, block freeze concentration technique was used to concentrate skim goat milk until three stages. The effects of freeze concentration on skim goat milk properties were evaluated by the analysis of total solids content, lactose content, total protein content, casein content, whey protein content, mineral content, and density. The color parameters of concentrate and ice fractions were also evaluated according to the CIELab color scale, and according to the rheological parameters. As the freeze concentration stages progressed, the total solids content, total protein, casein, and whey protein contents increased in both concentrate and ice fractions. In all stages, it was possible to note that the lactose content showed an equilibrium between both fractions. The densities values of both fractions also increased by increasing of the freeze concentration stages. Block freeze concentration obtained concentrates from skim goat milk with a whiteness index similar to whole milk. Overall, all concentrate and ice fractions showed tendency a greenish and yellowish color. The transition from Newtonian to non-Newtonian behavior was observed for concentrates and ices from second and third stages, respectively. Power Law and Herschel-Buckley models fitted to describe the behavior of the flow of all concentrate and ice fractions. The results generated in this study showed that concentrates from stage 1 and 2 demonstrated a promising product to be used by dairy industries.

KEYWORDS: Skim goat milk; block freeze concentration; goat milk concentrate; physical-chemical properties; rheological properties.

1. INTRODUCTION

The production of goat milk and its processing constitutes an economic activity of increasing importance due to the high nutritional interest of goat milk. Although goat milk production has been relatively minor compared to bovine milk, the world production of goat milk increased 17% between the years 2000 and 2016 [1]. Goat milk and its products are important in human nutrition and have become a part of the current trend of healthy eating around the world [2, 3, 4]. Besides that, the increase in demand for new dairy products with high added value in sophisticated market niches has stimulated goat milk production and trade [5]. The goat milk has high added value because it is a source of proteins of excellent quality, due to the proportion of essential amino acids [4, 6]. The importance of this milk is also intensifying because utilization of bovine milk had become a common cause of human food allergy [7]. The difference in protein composition of the goat milk in relation to cow milk, particularly regarding the casein fractions, made the goat milk be considered less allergenic [8, 9] and more

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digestible [7, 10, 11] in comparison with the cow milk. The goat milk has some particular properties that confer technological advantages with others species. The lower content of αs1-casein in the goat milk results in softer gel products, with a higher water holding capacity and with a lower viscosity [12].

The high nutritional value of goat milk throughout the world, including Brazil, has generated a need for a variety of techniques to preserve or increase these properties. According to Clark and Garcia [13]these studies have increased because of interest in intensive dairy goat production and value-added goat milk. However, emerging technologies, such as the freeze concentration process, is still innovative for goat milk. Even though that this process has the advantage of low energy usage with an effective concentration, for the goat milk industry, application of this process has been limited due to the dominance of more traditional technologies. However, it is known that industrial processes, such as heat treatment, homogenization, concentrating and spray drying, applied to goat milk can also affect their properties. However, the submission of goat milk to the freeze concentration process, in order to evaluate its effect on the properties of this milk specie, is innovative.

In the case of the block freeze concentration technique, the whole sample is frozen once as an ice block and then thawed slowly by gravity. One of the main advantages of this technique is related to the absence of moving parts (stirrers, pumps), which makes it a promising technology in relation to their operating costs. This technique is highlighted studies focusing on improving the performance in solutes recovery but not for goat milk. According to Petzold et al. [14] this process makes it possible to produce food concentrates with high quality by recovering a food solute based on the separation of pure ice crystals from a freeze-concentrate aqueous phase. When compared with traditional concentration processes, such as evaporation, the freeze concentration not only shows some significant potential advantages for the production of a concentrate where no vapor/liquid interface exists, but also can protect thermally fragile food compounds [14]. According to Sánchez et al. [15], the freeze concentration reduces around three times the total cost off the process (including capital, cleaning and energy), when compared to the evaporation or reverse osmosis processes. The concentration of solutes retained in the ice formed determines the efficiency of this process [16]. The freeze concentration process has already been used successfully by our research group for other products such as whey [17], milk [18], yerba mate extract [19], and tofu whey [20]. But additional research into methods to generate new products from goat milk will be essential in management of the global dairy industry. In this sense, the purpose of the present study was to evaluate the physicochemical and rheological properties of skim goat milk submitted to the block freeze concentration process.

2. MATERIALS AND METHODS

Material

Skim goat milk (Caprilat®, CCA Laticínios, Rio de Janeiro, Brazil) (8.46 ± 0.01 g total solids 100 g-1, 2.91 ± 0.05 g total protein 100 g-1, 3.93 ± 0.05 g lactose 100 g-1 and 0.89 ± 0.03 g ash 100 g-1) was used as feed material. All reagents were of analytical grade.

Protocol of the skim goat milk freeze concentration procedure

The freeze concentration procedure used to concentrate the skim goat milk was carried out by applying the block freeze concentration technique, according to the process proposed by Canella et al. [17]. An initial volume of 5 L of skim goat milk was separated into five batches of 1 L. Each batch of skim goat milk was fractionated in plastic containers and they were frozen at - 20 ± 2 °C in a freezer unit (Consul, Biplex CRD41D, São Bernardo do Campo, Brazil). After the skim goat milk has been completely frozen, 50 % of the initial volume was defrosted at room temperature (20 ± 2 °C), obtaining two fractions, the concentrate goat milk (CG1) and the ice (I1). The defrosted liquid that is the concentrate goat milk (CG1) was frozen again at - 20 ± 2 °C, and used as feed solution in the second stage. This procedure was repeated until the third stage. After each stage, a portion of concentrate (CG1, CG2, and CG3), and ice fractions (I1, I2 and I3) was collected and stored at - 20 ± 2 °C until physical, chemical and rheological analysis.

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Physicochemical analysis

The skim goat milk, concentrates (CG1, CG2, and CG3), and ice (I1, I2 and I3) fractions were analyzed in relation to the following physicochemical analysis: pH measurement; total solids content; lactose content; protein content (total protein, true protein, casein and whey protein content); and ash and mineral fractions.

The measurements of pH were obtained using a pH meter (PHS-3 BW, BEL, Piracicaba, São Paulo, Brazil). The total solids content (g 100 g^{-1}) were analyzed through the drying of the samples until reaching a constant weight at 103 ± 2°C and the ash content (g 100 g^{-1}) were analyzed through a gravimetric method [21].

The lactose content (g 100 g^{-1}) were obtained using a spectrophotometer FT-NIR model MPA (Multi Purpose Analyzer) (BrukerOptik, Ettlingen, Germany) operating with a spectral acquisition program OPUS version 7.0 (BrukerOptik, Ettlingen, Germany). The measurements were made by near-infrared Fourier transform (FTNIR) spectra of diffuse reflectance. Each vial containing the samples was positioned in the diffuse reflectance accessory and the NIR spectra were collected in the spectral range of 9.000 to 4.000 cm^{-1} at a nominal resolution of 16 cm^{-1} in transmission mode. Each spectrum was the average of 500 scans.

The total protein, true protein, casein and whey protein content (g 100 g^{-1}) were analyzed by the Kjeldahl method (N x 6.38) [22]. The determination of true protein was performed by Kjeldahl method, after previous preparation of samples, using the trichloroacetic acid at 15% for coagulation of all milk proteins that were removed by filtration, and the filter paper with the proteins coagulated was submitted to analyses.The fractions of milk casein were determined by the precipitation of milk casein of the samples in pH = 4.6 using acetic acid and sodium acetate solution. After precipitation, casein was separated by filtration and determined by Kjeldahl method. The whey protein was determined through the subtraction of casein content from the true protein.

Density

The density of skim goat milk, concentrates (CG1, CG2, and CG3), and ice (I1, I2 and I3) fractions was determinate by methodology described by AOAC [22]. A glass pycnometer (Gay-Lussac's pycnometer) (previously equilibrated to constant weight at 25 °C) was used. The density (g mL^{-1}) realized in triplicate was calculated using the following Equation 1:

$$\rho_S = \frac{(m3-m1)}{(m2-m1)x\rho H_2O} \qquad (1)$$

whereρ_S is the density of solutions (g mL^{-1}), m1 is the mass of empty pycnometer (g), m2 is the mass of pycnometer with water (g), m3 is the mass of pycnometer with samples (g), and ρH_2O is the density of water.

Color analysis

The color of the skim goat milk, and all fractions obtained by the freeze concentration process (CG1, CG2, CG3, I1, I2, and I3) were determined using a colorimeter Minolta Chroma Meter CR-400 (Konica Minolta, Osaka, Japan). The colorimeter was calibrated with a white standard plate and adjusted to operate with D65 lightning and 10º of observation angle. The CIELab color scale was used to measure the L^*, b^* and a^* parameters, that indicates the luminosity (variation from black to white), variation from yellow (+ b^*) to blue (- b^*) and variation from red (+ a^*) to green (- a^*), respectively. The total difference of color (ΔE) between the measured values of skim goat milk and each concentrates (CG1, CG2, and CG3), and ice (I1, I2 and I3) fractions was calculated according to Okpala et al. [23], as described in Equation 2,

$$\Delta E = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \quad (2)$$

where $\Delta L^*$is the difference of luminosity between the measured values of skim goat milk and each concentrates (CG1, CG2, and CG3), and ice (I1, I2 and I3) fractions, while Δa^*represents the intensity of the red color and Δb^* the intensity of the yellow color. The value of Hue angle (h^o) and Chroma (C^*) were determined using Equation 3 and 4, respectively. Five replicates were performed for each sample, and the mean values were reported.

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$h^* = tan^{-1}(b^*/a^*)$(3)

$C^* = \sqrt{(a^*)^2 + (b^*)^2}$(4)

Freeze concentration parameters

The freeze concentration parameters calculus was evaluated by concentration factor (CF), process efficiency (*eff*), and validations of results. CF was calculated in agreement with the method suggested by Aider and Ounis[24]. The CF of each freeze concentration stage was determinate as a function of the increase of total solids content, using the following Equation 5:

$$CF(\%) = \frac{TS_n}{TS_0} \, x \, 100 \qquad (5)$$

whereTS_n is the total solids (g 100 g^{-1}) content of the concentrate goat milk from each freeze concentration stage and TS_0 is the total solids (g 100 g^{-1}) content of the initial goat milk.

The process efficiency (*eff*) was calculated based on the increase of total solids (TS) in the concentrate goat milk (g 100 g^{-1}) relative to the TS remaining in the ice (g 100 g^{-1}) from each freeze concentration stage (n), as described in the Equation 6:

$$PE\ (\%) = \frac{\text{TS in the CG}_n - TS \text{ in the I}_n}{\text{TS in the CG}_n} \, x \, 100 \qquad (6)$$

To validate the experimental results obtained, the experimental mass balance of each stage was calculated and compared to the theoretical value [15] using the following Equation 7:

$$w_{pred} = \frac{\text{Ci}-\text{Cc}}{\text{Cg}-\text{Cc}} \qquad (7)$$

where *Wpred* is the predicted ice mass ratio (kg ice/kg skim goat milk), Ci is the total solids content of initial skim goat milk (g 100 g^{-1}), Cc is the total solids content of the concentrate fraction (g 100 g^{-1}) and Cg is the total solids content of the ice fraction (g 100 g^{-1}).

The root mean square deviation was calculated from Equation 8 to determine the deviation between experimental and theoretical data.

$$RSM\ (\%) = 100\sqrt{\frac{\sum\left(\frac{w_{exp}-w_{pred}}{w_{exp}}\right)^2}{N}} \qquad (8)$$

whereWexp and Wpred are the ratio of experimental and predicted ice mass, respectively, and N is the number of test repetitions.

Rheological analysis

The measurements of rheological properties of skim goat milk, concentrates (CG1, CG2, and CG3), and ice (I1, I2 and I3) fractions were carried out using a Thermo Haake DC 10 rotational viscosimeter (model VT 550, Thermo Haake, Karlsruhe, Germany), with concentric cylinders (NV ST 807-0713 CE and NV 807-0702), and collected using the software program Pro Rheowin® (version 2.93, Haake). Experimental studies were conducted on skim goat milk, concentrate (CG1, CG2, and CG3), and ice fraction (I1, I2, and I3) under controlled temperature at 5 °C. The control of temperature was realized through water circulation in a temperature controlled bath (Phoenix P1, Thermo Haake, Karlsruhe, Germany) and coupled to the equipment. An aliquot volume of 10 mL of samples was loaded into the cup of viscometer and the data were obtained. The flow curves were generated by a linearly increased shear rate of 0 s^{-1} to 2000 s^{-1} (upward curve) and 2000 s^{-1} to 0 s^{-1} (downward curve) during 3 minutes. To accurately evaluate the most adapted flow behavior, the models most frequently employed in food characterization [25] were used to describe the shear rate–shear stress data expressed by Equations 9 and 10.

$$\text{Power} - \text{law:}\quad \sigma = K(\dot{\gamma})^n \qquad (9)$$

$$\text{Herschel} - \text{Bulkley:}\quad \sigma = \sigma_0 + K\dot{\gamma}^n \qquad (10)$$

whereσ is shear stress (Pa), $\dot{\gamma}$ is shear rate (s^{-1}), K is consistency index (Pa s^{-1}), n is flow behavior index, and σ_0 is yield stress (Pa).

Statistical analysis
The data were expressed as means and standard deviations. One-way analyses of variance (ANOVA) and Tukey'sstudentized range (5 % significance) were carried out to test for any significant differences between the results. The validity of the results was evaluated based on the coefficient of determination (R^2). The data also were submitted to linear correlation (R) from regression analysis. All statistical analyses were performed using the software STATISTICA 13.3 software (TIBCO Software Inc., Palo Alto, CA).

3. RESULTS AND DISCUSSION

Physical and chemical parameters
The freeze concentration of skim goat milk resulted in a change of pH (Fig. 1). The concentrate fractions (CG1, CG2, and CG3) showed lower pH values (P< 0.05) than initial skim goat milk. In addition, with the evolution of the stages the pH values decreased (P< 0.05) in both fractions. Balde and Aider [26] noted the freeze concentration process of skim milk resulted in a slightly lower pH in concentrate fractions compared to pH of the fractions non-concentrate.

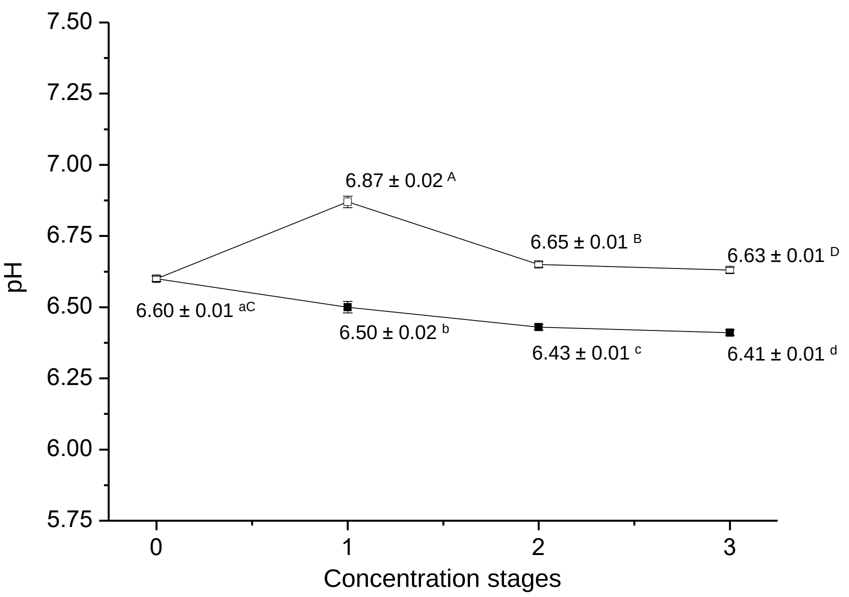

Figure1. Evolution of the pH in the concentrated (■) and ice (□) fraction as function of the freeze concentration stage from skim goat milk

The total solids contents of the initial skim goat milk, concentrates (CG1, CG2, and CG3), and ice fractions (I1, I2, I3) are showed in Figure 2. By increasing the freeze concentration stages, the total solids content in the concentrates fractions increased (P< 0.05) almost linearly presenting a correlation coefficient of R^2 = 0.9936.

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The concentrate goat milk reached values observed represent an increase of total solids content of 174, 226, and 284% at the first, second, and third freeze concentration stages, respectively, compared with the initial total solids content. These results are in agreement with those reported by Aider and Ounis [24] and Balde and Aider [26]during freeze concentration of skim cow milk. During the freeze concentration process, the total solids content of I1 and I2 decreased ($P< 0.05$) in comparison with initial skim goat milk. However, in the third stage, the total solids content in I3 increased significantly ($P< 0.05$). According to Aider and Ounis [24], this behavior is common, and could be associated to the presence of high total solids content. This is because when ice crystals are formed according to a hexagonal structure, the concentration of solution total solids is increased in the interstitial region between the growing ice crystals, which difficult the elimination of solids from the ice fraction [24, 27].

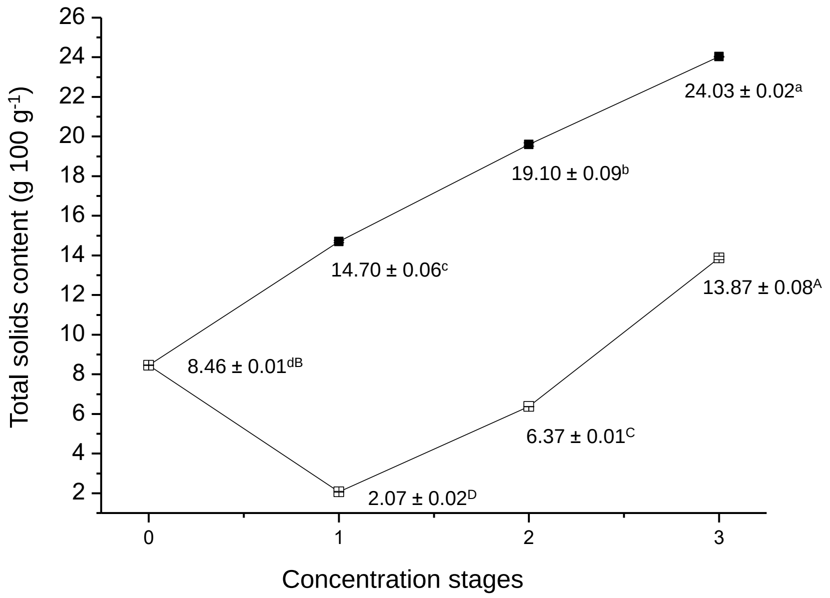

Figure 2. Evolution of the total solids content (g 100 g^{-1}) in the concentrated (■) and ice (□) fraction as function of the freeze concentration stages of skim goat milk.

The ash content and the mineral fractions of skim goat milk, concentrate (CG1, CG2, and CG3), and ice fractions (I1, I2, I3) are shown in Figure 3. In both concentrate and ice fraction, the ash content evolution showed similar behavior to that observed in the total solids content (Fig. 2). The values founded to ash content in concentrate fraction were higher than the ash content reported by Balde and Aider [26]during freeze concentration of skim cow milk. Ceballos et al. [28], Raynal-Ljutovac et al. [29], and Yadav et al.[30]credit this behavior to the fact that the goat milk contains mineral contents, as for example, such as Ca, P, K, Mg, at higher levels than the cow milk.

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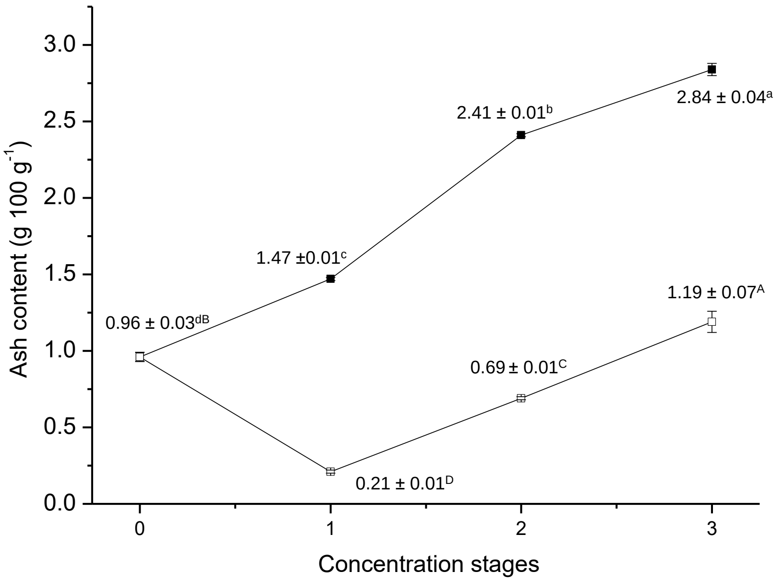

Figure 3. Evolution of the ash content (g 100 g^{-1}) in the concentrated (■) and ice (□) fraction as function of the freeze concentration stages of skim goat milk.

Lactose content of skim goat milk, concentrate (CG1, CG2, and CG3), and ice fractions (I1, I2, and I3) are shown in Figure 4. In the concentrate fractions, it was observed that the lactose content presented a slight increase (P< 0.05) in CG1 and CG2. At the third freeze concentration stage, lactose content of CG3 not showed (P> 0.05) difference with the CG2. In the ice fraction, lactose content followed different behaviors depending on the freeze concentration stage. The lactose content of I1 increased (P< 0.05) compared to its content in the initial skim goat milk. At the second freeze concentration stage, lactose content decreased (P< 0.05) presenting similar values to the initial skim goat milk. However, at the third stage of freeze concentration, lactose content in the I3 increased slightly (P< 0.05) again. Aider and Ounis [24] reported that when freeze concentration is used, the increase of the total solids content in the concentrate phase is accompanied by an increase in the amount of lactose entrapped in the ice crystals. Although the total solids content increased with the increase of freeze concentration stages, the lactose content was not accumulated in the only one fraction, once it is water soluble. Indeed, there were variations of the lactose content in the concentrate and ice fractions of skim goat milk, however, the values remained close to the lactose content of the initial skim goat milk. These results suggest that the concentrate fractions of skim milk goat, can be used in the manufacture of lactose intolerant products without increasing their costs with the extra addition of lactase enzymes.

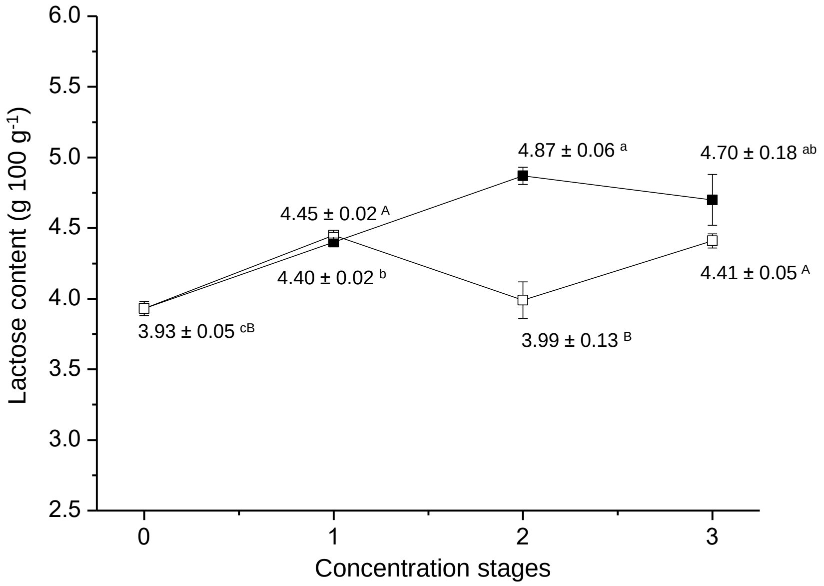

Figure 4.Evolution of the lactose content (g 100 g^{-1}) in the concentrated (■) and ice (□) fraction as function of the freeze concentration stages of skim goat milk.

Table 1 exhibits the total protein, casein, and whey protein content of skim goat milk, concentrate (CG1, CG2, and CG3), and ice fractions (I1, I2, I3). The results obtained showed the freeze concentration stages had a significant effect on total protein, casein, and whey protein content in the concentrate and ice fractions ($P < 0.05$). The increase of the total proteins content in the concentrate fraction followed a quasilinear kinetics (R^2=0,994). The results obtained in the present study are also in agreement to the results observed by Aider and Ounis (2012) and Balde and Aider [26], in skim cow milk freeze concentrate. However, the total protein content of CG2 and CG3 was higher than total protein content observed by Moreno-Montoro et al. [31] and Domagala and Kupiec [32] in skim goat milk and raw goat milk concentrate by ultrafiltration, respectively. In the ice fractions, total protein content decreased (P< 0.05) in the I1 and I2 when compared with the total protein content of initial skim goat milk. Thereafter, an increase (P< 0.05) in total protein content was observed in I3. The high concentration of total solids in this fraction (Fig. 2) was favorable to high protein inclusion in the I3.

Table 1. Total protein, casein and whey protein content of skim goat milk, concentrated (CG1, CG2, and CG3), and ice (I1, I2, and I3) fractions.

	Samples	Total protein (g 100 g^{-1})	Casein (g 100 g^{-1})	Whey protein (g 100 g^{-1})
	Skim goat milk	2.91 ± 0.05^{dB}	1.98 ± 0.01^{dB}	0.63 ± 0.10^{dB}
Stage 1	CG1	5.14 ± 0.02^{c}	3.18 ± 0.05^{c}	1.11 ± 0.05^{c}
	I1	0.72 ± 0.01^{D}	0.49 ± 0.01^{D}	0.17 ± 0.01^{C}
Stage 2	CG2	6.93 ± 0.01^{b}	4.47 ± 0.05^{b}	1.88 ± 0.17^{b}
	I2	2.20 ± 0.03^{C}	1.35 ± 0.01^{C}	0.54 ± 0.01^{B}
Stage 3	CG3	8.53 ± 0.02^{a}	5.32 ± 0.22^{a}	2.43 ±0.18^{a}
	I3	5.02 ±0.01^{A}	2.72 ± 0.03^{A}	1.03 ± 0.09^{A}

^{a,b,c} Within a column, means ± standard deviations with different superscript lowercase letters denote significant differences ($P < 0.05$) between the skim goat milk and the concentrated fraction of each freeze concentration stage. ^{A,B,C} Within a column, means ± standard deviations with different superscript uppercase letters denote significant differences ($P < 0.05$) between the skim goat milk and the ice fraction of each freeze concentration stage. CG1: concentrated fraction of first freeze concentration stage. I1: ice fraction of first freeze concentration stage. CG2: concentrated fraction of second freeze concentration stage. I2: ice fraction of second freeze

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concentration stage. CG3: concentrated fraction of third freeze concentration stage. I3: ice fraction of third freeze concentration stage.

Casein and whey protein content showed the same behavior of protein content, presenting an increase (*P*< 0.05) in the concentrate fractions (CG1, CG2, and CG3) with the increase of freeze concentration stages. As observed in total protein data, the casein and whey protein content decreased in the I1 and I3, when compared with the casein and whey protein content of initial skim goat milk, and only I3 presented casein and whey protein content higher than initial skim goat milk. According to Anema [33] and Balde and Aider [26], the volumetric fraction of the suspended material is determined mainly by proteins such as casein micelles, dissociated caseins, native whey proteins and denatured whey proteins.

Density

As observed in total solids content, by increasing the freeze concentration stages, the densities of concentrate (CG1, CG2 and CG3) increased (*P*< 0.05),since the total solids content are directly related to the density of a solution (Fig. 5). The densities of I1 and I2 also decreased (*P*< 0.05) in relation to density of initial skim goat milk (1.046 ± 0.001 g mL^{-1}). With the concentration change of the ice fraction, the milk density decreases as the amount of solvent increases and the amount of solute (milk components) remains constant or decreases. In addition, the increase of density in I3 (*P*< 0.05) corroborated with the increase of total solids content of this ice fraction. These results are in good agreement with those reported by Aider et al. [34]from studies with the freeze concentration of milk whey.

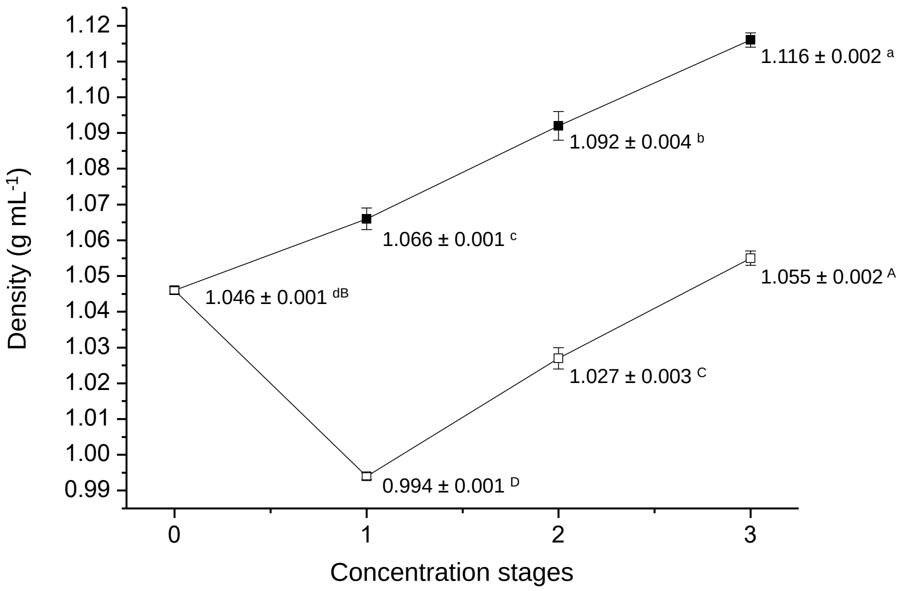

Figure 5.Evolution of the density (g mL⁻¹) in the concentrated (■) and ice (□) fraction as function of the freeze concentration stages of skim goat milk.

Color measurements

The Table 2 shows the color parameters of the initial skim goat milk, concentrate goat milk (CG1, CG2, and CG3), and ice fractions (I1, I2, and I3). As it can be observed on the data, the whiteness (*L**) increased (P < 0.05) until the second stage, with a small decrease at the third stage. Only the I3 showed higher (P < 0.05) whiteness than the initial skim goat milk. This behavior could be due to the total solids content in the final ice fraction. The values of L* determined in this study for concentrate fraction was higher than observed by Balde and Aider [26] for skim cow milk freeze concentrate. The reason for goat milk to be whiter than bovine milk is that goats convert all β-carotene into Vitamin A. According to Bermúdez-Aguirre et al. [35], in addition to

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nutritional properties, sensorial characteristics in milk, such as color, are very important for consumer acceptance. The whiteness (*L**) of milk has been shown to have a positive influence on consumer preference, which is why consumers have the more attractive for blades with visual properties close to the whole milk [35, 36].

Table 2. Color parameters of skim goat milk, concentrated (CG1, CG2, and CG3), and ice (I1, I2, and I3) fractions

Samples		Color parameters					
		*L**	*a**	*b**	*C**	*h**	Δ*E*
Skim goat milk		73.5 ± 0.25^{cB}	-3.22 ± 0.03^{bB}	4.19 ± 0.01^{dB}	5.28 ± 0.02^{dB}	127.55 ± 0.28^{aC}	-
Stage 1	CG 1	77.89 ± 0.25^{b}	-3.23 ± 0.01^{b}	7.64 ± 0.01^{c}	8.30 ± 0.02^{c}	112.88 ± 0.04^{b}	5.54 ± 0.10^{c}
	I1	56.27 ± 0.06^{D}	-2.11 ± 0.02^{A}	-3.20 ± 0.01^{D}	3.84 ± 0.01^{C}	236.63 ± 0.23^{A}	18.71 ± 0.05^{A}
Stage 2	CG 2	78.66 ± 0.07^{a}	-3.02 ± 0.01^{a}	10.27 ± 0.01^{b}	10.71 ± 0.01^{b}	106.41 ± 0.05^{c}	8.03 ± 0.05^{b}
	I2	68.26 ± 0.10^{C}	-3.54 ± 0.03^{C}	1.02 ± 0.03^{C}	3.69 ± 0.02^{D}	164.28 ± 0.14^{B}	6.06 ± 0.08^{B}
Stage 3	CG 3	77.85 ± 0.12^{b}	-3.37 ± 0.05^{c}	11.42 ± 0.03^{a}	11.91 ± 0.04^{a}	106.47 ± 0.19^{c}	8.46 ± 0.07^{a}
	I3	74.53 ± 0.13^{A}	-3.51 ± 0.03^{C}	6.20 ± 0.03^{A}	7.12 ± 0.03^{A}	119.55 ± 0.24^{D}	2.31 ± 0.08^{C}

a,b,c Within a column, means ± standard deviations with different superscript lowercase letters denote significant differences (P < 0.05) between the skim goat milk and the concentrated fraction of each freeze concentration stage. A,B,C Within a column, means ± standard deviations with different superscript uppercase letters denote significant differences (P < 0.05) between the skim goat milk and the ice fraction of each freeze concentration stage. CG1: concentrated fraction of first freeze concentration stage. I1: ice fraction of first freeze concentration stage. CG2: concentrated fraction of second freeze concentration stage. I2: ice fraction of second freeze concentration stage. CG3: concentrated fraction of third freeze concentration stage. I3: ice fraction of third freeze concentration stage.

All concentrate and ice fractions presented negative values for the *a** parameter, indicating a tendency to greenish color. However, this tendency was higher for CG3 and I3. According to Nozière et al. [37]*a** color parameter of the milk could be influenced by the natural pigment concentration presented in milk, such as the riboflavin. This pigment was a green compound present in the aqueous phase which is found in significant quantities in goat milk [38].

The initial skim goat milk and the concentrate fractions showed increase (P < 0.05) of *b** parameter, indicating a tendency for yellowish color with the increase of freeze concentration stages. According to Quiñones et al. [39] and Balde and Aider [26], the increase of the yellowness of milk is associated with the increase of protein content. This trend was confirmed by the protein determination presented in Table 1, where only the I1 showed negative values for *b**, and a tendency to the bluish color, due to the low protein content in this fraction. This parameter behavior also confirm the fact verified for the angle hue behavior.

As the Chroma C* parameter indicates the degree of saturation, purity or intensity of the color, it was observed that the C* of CG3 and I3 showed higher values (P < 0.05) than all the others fractions. Therefore, it was possible to note that, as the stages increased, the color intensified. The same behavior was observed to the Δ*E** value of concentration fractions, which increased (P < 0.05) with de increase of freeze concentration stages. At the same time, with the evolution of stages de total difference of color between ice fractions and skim goat milk decreased (P < 0.05), probably due to the total solids content in this fractions.

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Block freeze concentration performance

The performance of block freeze concentration procedure was determined from the total solids content obtained in concentrates and ice fractions. A progressive increase in concentration factor (CF) ($P < 0.05$) was observedwith the increase of freeze concentration stages (Fig. 6). This behavior is expected once the CF is directly related with the total solids, increasing its value with the increase of the total solids content. A similar performance was observed by Aider et al. [34], Aider and Ounis [24], and Muñoz et al. [18] in freeze concentration of cheese whey, skim milk, and whole milk, respectively.

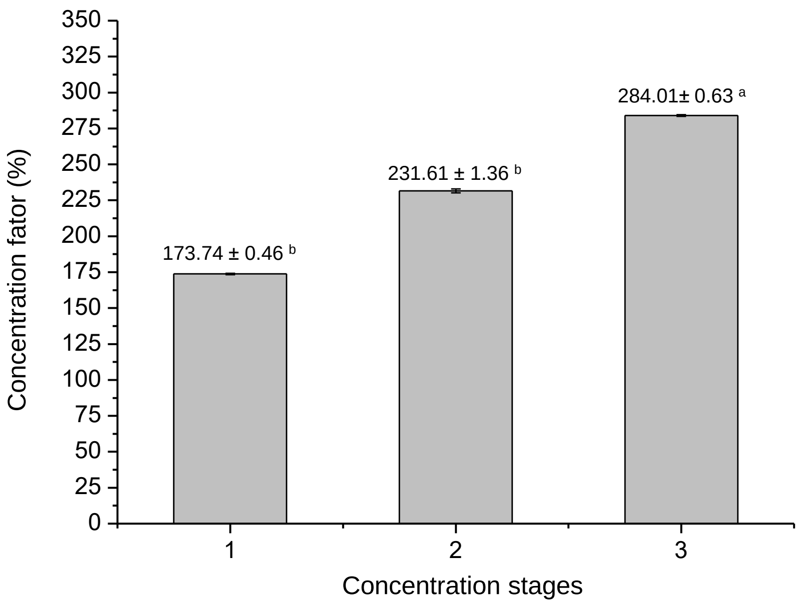

Figure 6. Evolution of concentration factor (CF) as function of the freeze concentration stages of skim goat milk

On the other hand, it was observed that the efficiency of the process (*eff*) decreases throughout the freeze concentration stages (Fig. 7). The same performance was noted by Aider et al. [40] and Balde and Aider [26]during freeze concentration of cheese whey and skim milk, where it was noted that *eff* is directly dependent on the total solids content in the ice fraction. Likewise, the density increase of I3 contributed to decrease of *eff*due to more solutes included in this fraction.

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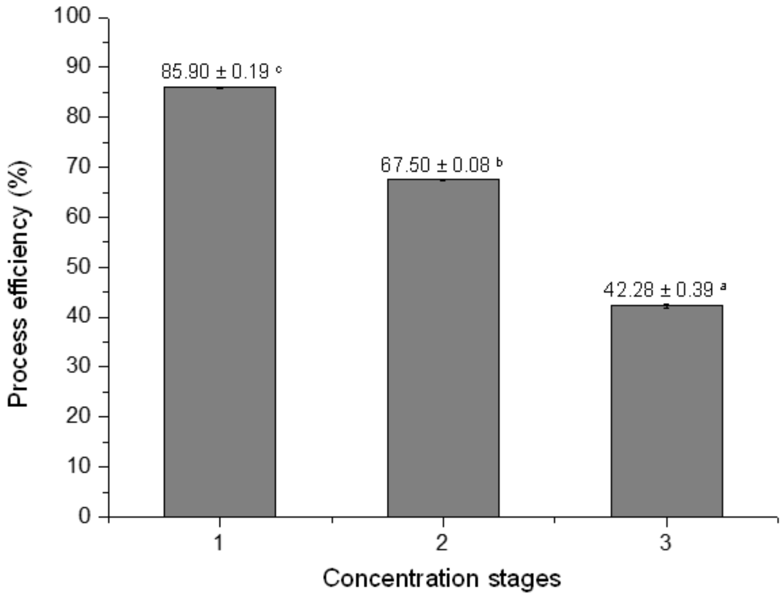

Figure7. Evolution of process efficiency (eff) as function of the freeze concentration stages of skim goat milk

When compared mass balance theorical data of each one freeze concentration stage with experimentally determined mass of the ice formed a good agreement is observed (Fig. 8). The root mean square deviation was calculated to determine the deviation between the experimental data and the theoretical estimates. It was observed a good adjustment of the process since the RSM values obtained in this study in the first (6.9%), second (12.0%), and third (12.2%) stage of concentration were lower than 25%, which is considered an acceptable fit, according to Lewicki [41]. These values were close to the values of 7.3%, 6.5%, 9.5%, and 10.8% reported by Hernández et al. [24], Petzold et al. [14, 43], and Belén et al. [44], respectively, in the freeze concentration process.

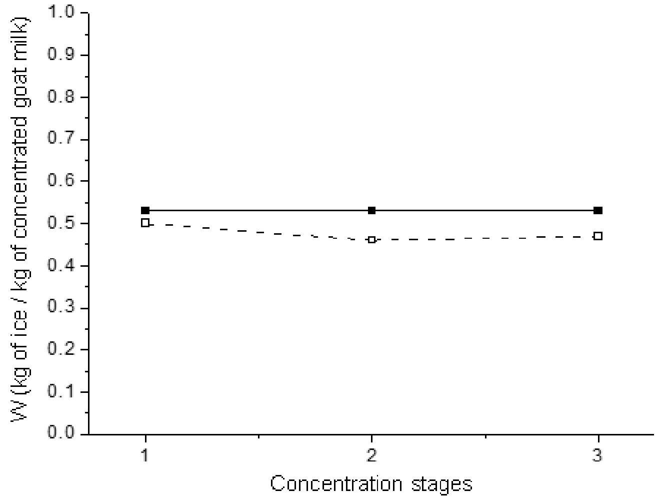

Figure8. Experimental (■) and predicted (- □ -) ice mass ratios as a function of freeze concentration stages of skim goat milk

Rheological analysis

In relation to the rheological parameters, as a function of shear rate profiles, the Figure 9 (a and b) shows relevant differences between the viscosity values of skim goat milk, concentrates (CG1, CG2, and CG3), and ice fractions (I1, I2, and I3). CG2, CG3, and I3 fractions showed a small decrease in apparent viscosity with increasing the shear rate, whereas the initial skim goat milk, CG1, I1, and I2 exhibited a Newtonian behavior. However, the apparent viscosity decreased in the CG2, CG3 and I3 fractions indicated that these fluids had shear thinning characteristics (non-Newtonian behavior). These results are in agreement with Vélez-Ruiz et al. [25] and Bienvenue et al. [45] who mentionated that the milk is a Newtonian fluid. According to Vélez-Ruiz and Barbosa-Cánovas [46], when a Newtonian nature is detected, this behavior is related to low solids concentrations, such as observed in this study in the fractions CG1, I1, and I2 (Figure 2).

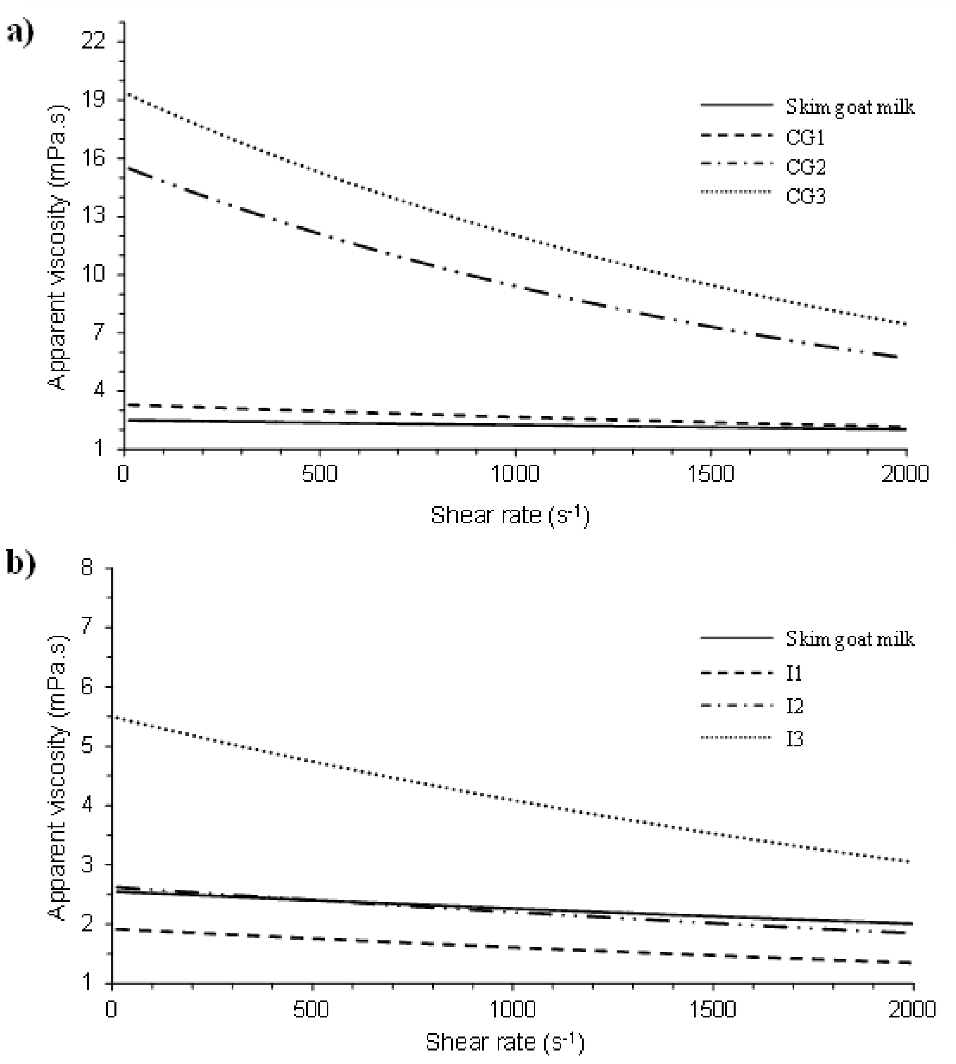

Figure9. Apparent viscosity versus shear rate of (a) skim goat milk and concentrated fraction (CG1, CG2, CG3), and (b) skim goat milk and ice fraction (I1, I2, I3)

It was also possible to note that the viscosity of all samples increases when concentration stages increased,which is also credited to increases in the total solids and in the protein content (Table 1). The same behavior was observed by Aider and Ounis [24] in skim milk freeze concentrate. According to Anema et al. [33], the viscosity of a system is dependent on the volume fraction occupied by the contributing particles in combination with the inherent viscosity of the continuous phase. In skim milk systems, the proteins determine the volume fraction of the suspended material. This increased of viscosity occurs because the removal of water causes an increase in volume fraction of dispersed particles and increases the micelle-micelle interactions as the distance between the micelles becomes smaller [45]. Also, it was reported that the ability to separate ice of high purity from the concentrate is dependent and inversely proportional to the solution viscosity [14, 15, 47], explaining the decrease in *eff* of this study (Fig. 7).

The rheological behavior of a fluid can be described with two values: the “consistency coefficient” K, and the “flow behavior index” *n*. The flow behavior index describes the shear behavior of the fluid and is a measure of the departure from Newtonian behavior. If the *n* value is close to 1, the sample is Newtonian. If *n* is less than 1, the sample shear thins with increasing shear rate, whereas if *n* is higher than 1, the sample shear thickens [33]. There are various models to describe the dependence of viscosity from shear rate and to determinate the consistency coefficient and the flow behavior index [48, 49]. In our study, different models were tested; however, the best adjustments were founded with the Power law and Herschel-Bulkley models. In accordance with Balde and Aider [26], Chang and Hartel [50], and Vélez-Ruiz and Barbosa-Cánovas [46] the milk concentrates behaved as non-Newtonian fluids, with flow curves well fitted by the Power law and/or Herschel-Bulkley models. Therefore, the fit for all data sets was good in both models, with a coefficient of determination between 0.93 and 0.97 (Table 3).

The flow parameters of the initial skim goat milk, concentrates goat milk (CG1, CG2, and CG3), and ice fractions (I1, I2, and I3) are in the Table 3. Although the Herschel-Bulkley model presented a good fit, the yield stress were of the samples were very small, close to 0. These results are in agreement with those obtained by Balde and Aider [26]. These authors affirmed that when the total solid content is lower than 30 g 100 g^{-1} the yield stress decrease. The initial skim goat milk, CG1, I1, and I2 exhibited a Newtonian behavior because *n* values were very close to 1, whereas the fractions CG2, CG3, and I3 showed *n* lower than 1, and therefore a shear thinning behavior. From the results obtained for the parameter *n* of both models it was possible to confirm the behavior of samples observed previously in Figure 9.

According to Power law and Herschel-Bulkleymodels it was noted that the consistency index was higher ($P<0.05$) in the final fractions of freeze concentration process (CG3 and I3). As expected, the consistency of concentrate goat milk increased with its total solids content (Fig. 2), and this behavior is in accordance with the results observed for other food concentrates such as skim cow milk [26], grape juice [51] and orange juice [52]. The increase in the consistency index of concentrates is justified by the sum of the interaction effects caused by each of the individual milk particles suspended in a medium with less water content [50]. The values of consistency index of skim goat milk founded in this study were higher than consistency index of freeze concentrate skim cow milk observed by Balde and Aider [26]. According to Park et al. [38], the viscosity of goat milk is slightly higher than in cow milk. Thereby, with these results we can highlight that the freeze concentration was an efficient technique for the skim goat milk concentration. We also recommend that the concentrates obtained up to the second stage can be used in the development of news goat milk products, which will contain even more valuable nutritional properties. It is hoped that the results of this study will allow a better understanding of the performance of skim goat milk freeze concentration, because the development of alternative technologies to protect and retain nutritional quality of goat milk are extremely relevant for use industrial.

Table 2. Color parameters of skim goat milk, concentrated (CG1, CG2, and CG3), and ice (I1, I2, and I3) fractions.

	Samples	Power Law model			Herschell-Buckleymodel			
		K (Pa.s^n)	*n*	R^2	σ_0	K (Pa.s^n)	*n*	R^2
	Skimgoatmil k	0.003 ± 0.001^{cB}	0.970 ± 0.021^{aA}	0.97^{a} AB	0.051 ± 0.018^{cA}	0.002 ± 0.001^{bB}	0.990 ± 0.030^{aAB}	0.97^{aA}
Stag e 1	CG1	0.003 ± 0.001^{cB}	0.957 ± 0.035^{a}	0.96^{a} bC	0.089 ± 0.034^{bc}	0.002 ± 0.001^{b}	1.013 ± 0.031^{a}	0.96^{ab}
	I1	0.002 ± 0.001^{D}	0.973 ± 0.021^{A}	0.95^{B}	0.024 ± 0.008^{A}	0.001 ± 0.001^{B}	1.043 ± 0.029^{A}	0.93^{B}
Stag	CG2	0.024 ±	0.833 ±	0.95^{b}	0.334 ±	0.015 ±	0.891 ±	0.95^{b}

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e 2		0.003^{b}	0.021^{b}		0.021^{ab}	0.001^{a}	0.004^{b}	
	I2	0.002 ± 0.001^{B}	0.968 ± 0.008^{A}	0.96^{B}	0.044 ± 0.053^{A}	0.002 ± 0.001^{B}	0.996 ± 0.027^{AB}	0.96^{A}
Stage 3	CG3	0.032 ± 0.002^{a}	0.831 ± 0.007^{b}	0.96^{a}	0.564 ± 0.212^{a}	0.019 ± 0.004^{a}	0.892 ± 0.018^{b}	0.96^{a}
	I3	0.006 ± 0.001^{A}	0.929 ± 0.017^{A}	0.97^{A}	0.110 ± 0.049^{A}	0.004 ± 0.001^{A}	0.963 ± 0.023^{B}	0.97^{A}

^{a,b,c} Within a column, means ± standard deviations with different superscript lowercase letters denote significant differences ($P < 0.05$) between the skim goat milk and the concentrated fraction of each freeze concentration stage.

^{A,B,C} Within a column, means ± standard deviations with different superscript uppercase letters denote significant differences ($P < 0.05$) between the skim goat milk and the ice fraction of each freeze concentration stage. K, Consistency index; n, flow behavior index; R^2, determination coefficient; σ_0, yield stress. CG1: concentrated fraction of first freeze concentration stage. I1: ice fraction of first freeze concentration stage. CG2: concentrated fraction of second freeze concentration stage. I2: ice fraction of second freeze concentration stage. CG3: concentrated fraction of third freeze concentration stage. I3: ice fraction of third freeze concentration stage.

4. CONCLUSION

Skim goat milk was successfully concentrate by applying the block freeze concentration procedure. The high total solids content presented by I3 explains the higher efficiency of the process achieved by the first and second stages. While de total solids content increased in both concentrated and ice fractions, the pH values reduced. The values of lactose content showed no difference at the end of second and third concentrate stage. The total proteins were also concentrate and their content reached up reached almost 3 times more than the initial total protein value of skim goat milk. As the total protein content, the casein and whey protein content increased with increasing freeze concentrate stages, in both concentrate and ice fractions. The concentrate and ice of third stage (CG3 and I3) presented the higher densities. In addition, it was also possible to obtain concentrates skim goat milk with a whiteness index similar to that of whole milk, as observed by L* value. In general, all the concentrates and ice fractions presented tendency a greenish and yellowish color. The Power Law and Herschel-Buckley models were fitting to describe the flow behavior for all concentrate and ice fractions. The transition from a Newtonian to a non-Newtonian behavior was observed for the second stage to concentrate fractions, and for the ice from third stage. The results obtained applying the block freeze concentration suggests that freeze concentration stages positively affected the skim goat milk. Finally, both concentrates from the first and from the second stages would be used for the manufacture of goat's milk products.

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