Performance of skim goat milk mineral content subjected to the block freeze concentration process

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Authors' contributions

This work was carried out in collaboration between all authors. M H M C and E S P designed this study, reviewed all steps and the data analysis. M H M C, L M and E S P wrote the protocol of the analysis and the first draft. M H M C, E L S B and C C S realized the statistical analysis and managed the literature searches. S V and H D reviewed all steps of this work. All authors read and approved the final manuscript.

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ABSTRACT

The aim of this study was to evaluate the goat milk mineral performance concentrated by block freeze concentration process. Twenty batches of skim goat milk, each one with one liter, were subjected until the third stage of the freeze concentration process. The initial skim goat milk, concentrated, and ice fractions obtained were analyzed by calcium, magnesium, zinc, phosphorus, sodium and potassium content. Results showed that by application of the freeze concentration only phosphorus content not increased (*P* < *0.05*), for concentrated and ice fractions.??? In the first stage of freeze concentration process, the magnesium element showed the higher *(P* < *0.05)* efficiency (95 %). However, the higher *(P* < *0.05)* concentration factor was determinate to calcium element in the third stage (10000 %). Also, it was observed an increase in the minerals contents evaluated with the increasing of freeze concentration stages of skim goat milk. Based on results obtained in the present study, the concentrated from the first stage showed the best performance of skim goat milk mineral content.

Keywords: Goat milk concentrated, concentration process, main mineral elements, efficiency of process, concentration factor.

1. INTRODUCTION

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 [1, 2]. Goat milk has high added value because it is a source of nutritional compositional of excellent quality, including the quantity of minerals [3, 4]. Goat milk present some major and minority minerals in larger amounts than cow milk [5-7]. Minerals are fundamental for human health, as they are required for many physiological functions such as tissue growth, regulation of enzyme activities, blood clotting, and to facility of membrane transport of essential nutrients [8, 9]. Besides their effects on health, minerals influence milk technological traits, casein micelle structure and aggregation, rennet coagulation time, curd structure, and cheese yield [8, 10-11].

It is known that most people consume foods that have less than two-thirds of one or more essential minerals [12]. In addition, because of that, the production of mineral-supplemented foods is growing as an important strategy to prevent mineral deficiencies. Milk and milk based products are good materials for mineral fortification due to their worldwide consumption by all groups at risk of deficiency, and also because of their high nutritional value, of the buffering effect on digestion and absorption processes, and of the positive effects on growth [9]. The concentration of milk may be an alternative to supplementation of these products. New methods are developed to increase goat milk and its derivate quality. Also, the development of new added value products has led to increased interest in specific studies focused on the suitable ways of improve goat milk nutrition, quality, and consumption.

The block freeze concentration technology makes it possible to produce food concentrated with high quality by recovering a food solute based on the separation of pure ice crystals from a freeze-concentrated aqueous phase. When compared with traditional concentration processes, such as evaporation, freeze concentration not only shows some significant potential advantages for the production of a concentrated where no vapor/liquid interface exists but also can protect thermally fragile food compounds [13]. According to Sánchez et al. [14], the freeze concentration reduces about three times the total cost of the process (including capital, cleaning and energy), when compared to the evaporation or reverse osmosis processes.

The freeze concentration has highly promising applications, especially, in the production of foods and ingredients that have high nutritive value [15]. In this technology, a food liquid solution is completely frozen and then, the whole frozen solution is thawed, with separation of concentrated fraction from ice fraction by gravitational thawing. Sometimes the separation may be carried out assisted by other techniques to enhance separation efficiency [16, 17]. The concentration of solutes retained in the ice formed determines the efficiency of this process [15]. This technique has been used in concentration of different foods, such as cheese whey [14, 18], milk [19], skim milk [20], wine [17], fruit juices [13, 21, 22], coffee extract [23], and tofu whey [24].

Studies have been conducted on the properties of concentrated skim goat milk prepared by ultrafiltration [11]. However, in the light of our knowledge, there are no reports in the literature on how mineral performance of skim goat milk is affected by the block freeze concentration technology. A better understanding of this behavior is necessary to further understand the use of freeze concentrated milk in production and processing of new dairy products. Therefore, the aim of the present study was to concentrate skim goat milk by block freeze concentration process and to evaluate the impact of the process on mineral performance of the concentrated and the ice fractions.

2. MATERIALS AND METHODS

2.1 Materials

Commercial skim UHT goat milk (Caprilat®, CCA Laticínios, Rio de Janeiro, Brazil) was used as the start material. The skim goat milk composition was 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}. All reagents were of analytical grade.

2.2. 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 [25]. An initial volume of 20 L of skim goat milk was separated into twenty batches of 1 L. Each 1L of skim goat milk was fractionated in plastic containers and 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 concentrated goat milk (CG1) and the ice (I1). The defrosted liquid (CG1) was frozen at - 20 ± 2 °C and used as feed solution in the second stage. This procedure was repeated until the third stage (Figure 1). After each stage, a portion of concentrated (CG1, CG2, and CG3), and ice fractions (I1, I2 and I3) was collected and stored at - 20 ± 2 °C until the analysis.

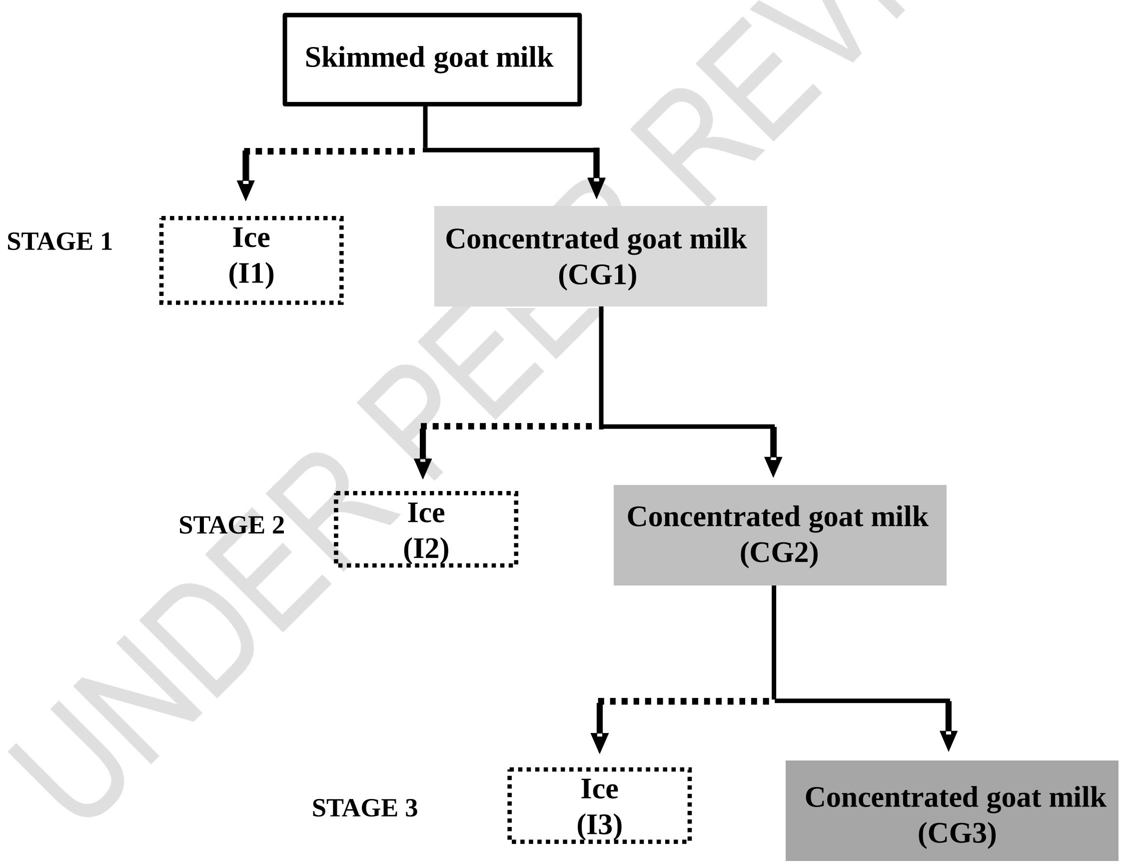

Fig. 1. Diagram of the skim goat milk block freeze concentration process.

2.3 Mineral fractions analysis

2.3.1 Calcium, magnesium, and zinc content

The determination of mineral elements Ca, Mg, and Zn content (mg kg^{-1}) were carried out by flame atomic absorption spectrometry (F-AAS) according to Navarro-Alarcón et al. [26], with modifications. The spectrometer used was the AAnalyst 200 model (PerkinElmer, Inc., Waltham, MA, EUA) equipped with the background corrector, and the deuterium arc illumination, using the Echelle resolution system. Acetylene (purity 99.7 %) was employed as fuel gas to heat the atomization system and as compressed gas was used as the compressed air. Before the measurement, all samples were calcined at 520 °C, and the ash obtained were treated with hydrochloric acid 8 mol L^{-1}. The analytical and instrumental parameters were adjusted to obtain the best sensitivity for each element (Table 1). For this, the samples were diluted with Milli-Q water for interpolation in the linear range of each mineral element. Cathode lamps (PerkinElmer, Inc., Waltham, MA, USA) were employed to determinate minerals elements. All analyses were carried out in triplicate, and blanks were prepared with bidistilled deionized water.

Table 1. Flame atomic absorption spectrometry (F-AAS) instrumental parameters.

Minerals	Wavelengths (nm)	Linear range (mg kg^{-1})
Ca	422.67	1.00 - 5.00
Mg	285.21	0.10 - 0.30
Zn	213.86	0.10 - 1.50

<u>2.3.2 Phosphorus content</u>

Phosphorus content (mg kg^{-1}) was measured by molecular spectrometry at 420 nm in a spectrophotometer UV-Vis, with deuterium lamp (Thermo Fisher Scientific Inc., Waltham, MA, USA). The samples were initially calcined (520 °C), and complexed with molybdenum phosphoric acid. Samples results were interpolated in calibration curves constructed with diacid phosphate of potassium, in the range of 1 to 20 mg L^{-1}. All analyses were carried out in triplicate, and blanks were prepared with bidistilled deionized water.

<u>2.3.3 Sodium and potassium content</u>

The sodium and potassium content (mg kg^{-1}) were determined through the technique of atomic emission spectrometry (F-AES), with a flame photometer 910M (Analyser Comércio e Indústria Ltda., São Paulo, Brazil) at 589.0 e 710 nm, respectively. For the evaluation of these minerals, the samples were calcined at 520 ° C, and treated with nitric acid 4 mol L^{-1}. Sample results were interpolated in calibration curves constructed in the range of 1 to 10 mg L^{-1}. All analyses were carried out in triplicate, and blanks were prepared with bidistilled deionized water.

2.4 Freeze concentration parameters

<u>2.4.1 Concentration factor</u>

The concentration factor (CF) was calculated in agreement with the method proposed by Aider and Ounis [27]. The CF of each freeze concentration stage was determinate as a function of the increase of mineral content, using the following Equation 1:

$$CF\ (\%) = \frac{MC_n}{MC_0}\ x\ 100 \qquad (1)$$

where MC_n is the mineral (mg kg^{-1}) content of the concentrated goat milk from each freeze concentration stage and MC_0 is the mineral (mg kg^{-1}) content of the initial skim goat milk.

2.4.2 Process efficiency

The process efficiency (*eff*) was calculated based on the increase of mineral content (mg kg^{-1}) in the concentrated goat milk (MC_n) in relation to the mineral content (mg kg^{-1}) remaining in the ice (MC_i) from each freeze concentration stage (n), as described in the Equation 2:

$$eff\ (\%) = \frac{MC_n - MC_i}{MC_n}\ x\ 100 \qquad (2)$$

2.5 Statistical analysis

Data were expressed as means and standard deviations. Statistical analysis of data was performed using the software STATISTICA 13.3 software (TIBCO Software Inc., Palo Alto, CA). One-way analyses of variance (ANOVA) and Tukey's studentized range (5 % significance) were carried out to test significant differences between the results.

3. RESULTS AND DISCUSSION

Goat milk is considered an exceptionally important food because is rich in mineral content. The mineral fractions of skim goat milk, concentrated (CG1, CG2, and CG3), and ice fractions (I1, I2, I3) are shown in Table 2. Generally, the mineral content in the concentrated and ice fraction increased with increase in freeze concentration stages. When verified the concentration of major elements such as Ca, Mg, Na, K and P, it was possible to note that the values of Ca, Mg, Na, K were higher ($P < 0.05$) in all concentrated fractions (CG1, CG2, and CG3), when compared with the initial skim goat milk. Besides that, these minerals contents in CG1, CG2, and CG3 increased ($P < 0.05$) with the increase of the freeze concentration stages. This performance over the freeze concentration stages was expected, because similar behavior was reported in block freeze concentration process of the skim cow milk [20]. The concentration of Ca and Mg were higher than those reported by Moreno-Montoro et al. [11] during the ultrafiltration of skimmed goat milk. Ca and Mg contents are related to casein structure, which is primarily involved in the coagulation process and curd formation, and a higher concentration of Ca to milk decreases rennet clotting time and increases curd firmess [10, 28-30]. The P content showed no difference ($P > 0.05$) between the initial skim goat milk and concentrated fraction (CG1, CG2, and CG3). It was noted a slight progressive increase in relation to Ca, Mg, Na, K, and P contents for the ice fractions of freeze concentration stages. However, I1 and I2 fractions showed lower values ($P < 0.05$) of these minerals when compared with the initial skim goat milk.

Table 2. Mineral contents performance of skim goat milk, concentrated (CG1, CG2, and CG3), and ice (I1, I2, and I3) fractions during block freeze concentration stages.

Samples	Ca (mg kg^{-1})	Mg (mg kg^{-1})	Na (mg kg^{-1})	K (mg kg^{-1})	P (mg kg^{-1})	Zn (mg kg^{-1})
Skim goat milk	987.48 ± 3.38^{dB}	82.52 ± 1.21^{dB}	676.59 ± 0.32^{dB}	1429.89 ± 108.48^{dB}	476.74 ± 35.62^{aB}	6.97 ± 0.03^{dA}
CG1	1720.38 ± 3.31^{c}	147.36 ± 0.01^{c}	1004.86 ± 1.19^{c}	1842.38 ± 239.03^{c}	508.25 ± 4.55^{a}	10.71 ± 0.01^{b}
I1	122.30 ± 0.01^{D}	7.11 ± 0.12^{D}	316.00 ± 6.00^{D}	316.02 ± 6.50^{D}	242.52 ± 5.96^{D}	0.93 ± 0.01^{D}
CG2	2307.57 ± 6.66^{b}	235.97 ± 1.20^{b}	1258.26 ± 0.56^{b}	2831.08 ± 1.26^{b}	503.99 ± 0.22^{a}	7.94 ± 0.03^{c}
I2	707.34 ± 6.52^{C}	13.97 ± 0.01^{C}	608.27 ± 0.52^{C}	1216.55 ± 1.05^{C}	420.83 ± 0.11^{C}	2.55 ± 0.01^{C}
CG3	10388.28 ± 213.62^{a}	458.99 ± 9.44^{a}	1652.70 ± 33.99^{a}	3305.39 ± 67.97^{a}	522.40 ± 10.74^{a}	17.36 ± 0.36^{a}
I3	$1494,75 \pm 6.67^{A}$	136.45 ± 1.20^{A}	880.67 ± 1.28^{A}	1907.97 ± 204.80^{A}	529.81 ± 0.17^{A}	5.26 ± 0.01^{B}

[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.

The Zn content decreased (*P* < 0.05) for the CG2 in comparison with the CG1, and with the skim goat milk. At the third stage, the Zn content increased (*P* < 0.05), showing higher values for the CG3. The initial skim goat milk showed higher (*P* < 0.05) Zn content than all ice fractions. According to Gao et al. [31], and Aider and Ounis [27], freezing of salt solution above its eutectic temperature causes rejection of salt (poorly soluble in ice) to the surrounding medium, creating water with very high salt content brine.

Minerals content of Ca, P, K, Na and Mg were higher than those reported by Balde and Aider [20] during the block freeze concentration of skim cow milk. This performance could be related to the fact the goat milk present some mineral contents, such as Ca, P, K and Mg, in larger amounts than cow milk [5-7].

Regarding mineral efficiency concentration (Fig. 2), overall notable values were achieved. However, the best value was obtained at concentration of Mg with an efficiency of approximately 95 % in the first stage and around 70 % at the third stage. The lowest efficiency was to P concentration with an efficiency of 52 %, 16 %, and 17 % at the first, second, and third stages, respectively. In general, the highest process efficiencies were recorded at the end of the first freeze concentration stages. These results indicate that more minerals were entrapped in the ice fraction at the final stages of freeze concentration process (I2 and I3). This performance was also stated by Aider, de Halleux, and Melnikova [32] for the freeze concentration of skim acidic milk.

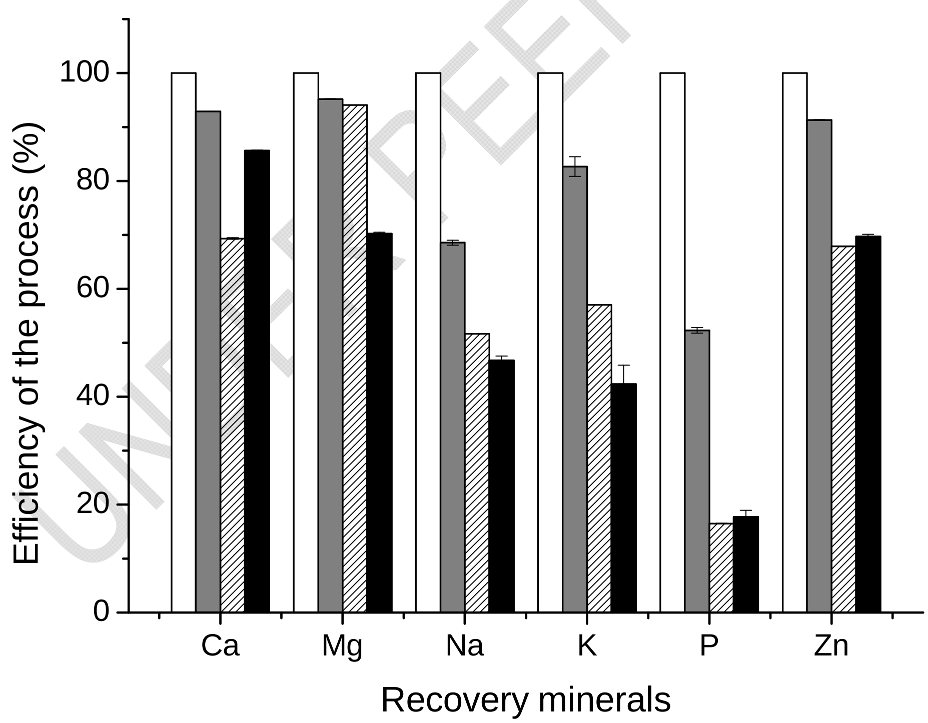

Fig.2. Performance of freeze concentration process on the efficiency (eff) of goat milk minerals concentration as a function of freeze concentration stages (□ initial skim goat milk, ▨ stage 1, ▨stage 2, and ■stage 3).

In the present study, for all mineral content evaluated, the concentration factor (CF) (Fig. 3) showed an opposite performance than those observed by the mineral efficiency concentration. An increase ($P < 0.05$) of the concentration factor was observed over the freeze concentration stages, reaching a CF of 10000 % for the Ca content in the third freeze concentration stage.

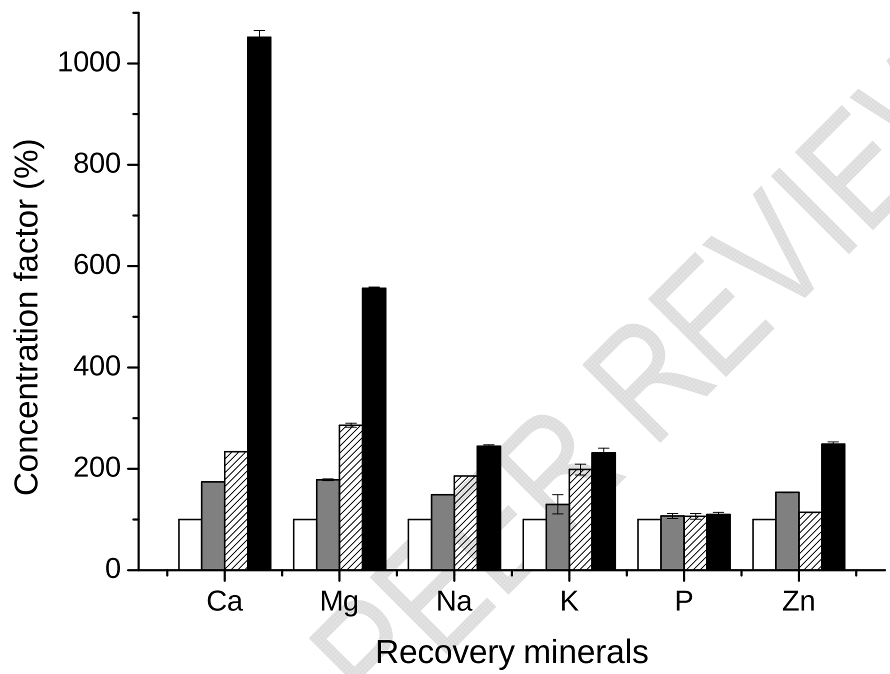

Fig.3. Performance of freeze concentration process on the concentration factor (CF) of goat milk minerals concentration as a function of freeze concentration stages (initial skim goat milk, stage 1, stage 2, and stage 3).

As observed by Ceballos et al. [5], Yadav, Singh, and Yadav [7], and Campos et al. [33] in the present work it is possible to note that main elements contents of skim goat milk are higher than cow milk. Finally, in a near future, the generate results from the block freeze concentration process of skim goat milk mineral content performance can be used by dairy industries to produce nutritive products with high mineral contents without mineral supplementation, which positively affects the economic and the nutritive value of milk products.

4. CONCLUSION

The mineral content of skim goat milk was successfully freeze concentrated by applying the block freeze concentration. As the freeze concentration stages increased, Ca, Mg, Na, K, and Zn contents increased in both concentrated and ice fractions. It was possible to concentrated Ca and Mg after three stages, around 10 and 6 times more than the initial skim goat milk, respectively. Indeed, the K, Na and Zn were concentrated after three stages, almost 3 times more than initial skim goat milk, respectively. However, the phosphorus showed no difference of concentrated fraction in the three stages compared with the initial skim goat milk. All mineral content showed high efficiency and concentration factor during the freeze concentration process. The study concluded that the concentrated from the first stage was the best. This is because higher efficiencies results were obtained in this stage.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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