

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

ABSTRACT - MAKE AS AIM, PLACE AND DURATION, DESIGN, RESULT, CONCLUSION

The goat milk possess good nutritional and health-promoting properties, and besides it is considered an exceptionally important food because is rich in mineral content, being also able to influence in the dairy technology. The aim of this study was to evaluate the goat milk mineral performance submitted to block freeze concentration process. Therefore, at the end of this study it was possible to note that with the freeze concentration stages increase, calcium, magnesium, sodium, potassium, and zinc contents increased in both concentrated and ice fractions. However, the phosphate content showed no difference between concentrated and the initial skim goat milk. All mineral content showed high efficiency of process and concentration factor. Finally, it was observed an increase in the minerals contents evaluated with the increasing of freeze concentration stages of skim goat milk. This behavior was highlighted for the results obtained to the efficiency of mineral concentration determined in the first stage of the freeze concentration process.

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]. The goat milk has high added value because it is a source of nutritional compositional of excellent quality, including the quantity of minerals [3, 4]. The 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 a growing as an important strategy to prevent mineral deficiencies. Milk and milk based products are good candidates 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

41 concentration processes, such as evaporation, freeze concentration not only shows some
42 significant potential advantages for the production of a concentrated where no vapor/liquid
43 interface exists but also can protect thermally fragile food compounds [13]. According to
44 Sánchez et al. [14], the freeze concentration reduces around three times the total cost off the
45 process (including capital, cleaning and energy), when compared to the evaporation or
46 reverse osmosis processes.

47 The freeze concentration has highly promising applications, especially, in the
48 production of foods and ingredients that have high nutritive value [15]. In this technology, a
49 food liquid solution is completely frozen and then, the whole frozen solution is thawed, with
50 separation of concentrated fraction from ice fraction by gravitational thawing. Sometimes the
51 separation may be carried out assisted by other techniques to enhance separation efficiency
52 [16, 17]. The concentration of solutes retained in the ice formed determines the efficiency of
53 this process [15]. This technique has been used in concentration of different foods, such as
54 cheese whey [14, 18], milk [19], skim milk [20], wine [17], fruit juices [13, 21, 22], coffee
55 extract [23], and tofu whey [24].

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

64 65 66 **2. MATERIAL AND METHODS**

67 68 **2.1 Materials**

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70 Commercial skim UHT goat milk (Caprilat®, CCA Laticínios, Rio de Janeiro, Brazil) was
71 used as the start material. The skim goat milk composition was 8.46 ± 0.01 g total solids 100
72 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
73 100 g^{-1} . All reagents were of analytical grade.

74 75 **2.2. Protocol of the skim goat milk freeze concentration procedure**

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77 The freeze concentration procedure used to concentrated the skim goat milk was carried out
78 by applying the block freeze concentration technique, according to the process proposed by
79 [25]. An initial volume of 20 L of skim goat milk was separated into twenty batches of 1 L.
80 Each 1L of skim goat milk was fractionated in plastic containers and were frozen at -20 ± 2
81 $^{\circ}\text{C}$ in a freezer unit (Consul, Biplex CRD41D, São Bernardo do Campo, Brazil). After the
82 skim goat milk has been completely frozen, 50 % of the initial volume was defrosted at room
83 temperature (20 ± 2 $^{\circ}\text{C}$), obtaining two fractions, the concentrated goat milk (CG1) and the
84 ice (I1). The defrosted liquid (CG1) was frozen at -20 ± 2 $^{\circ}\text{C}$ and used as feed solution in
85 the second stage. This procedure was repeated until the third stage (Figure 1). After each
86 stage, a portion of concentrated (CG1, CG2, and CG3), and ice fractions (I1, I2 and I3) was
87 collected and stored at -20 ± 2 $^{\circ}\text{C}$ until the analysis.

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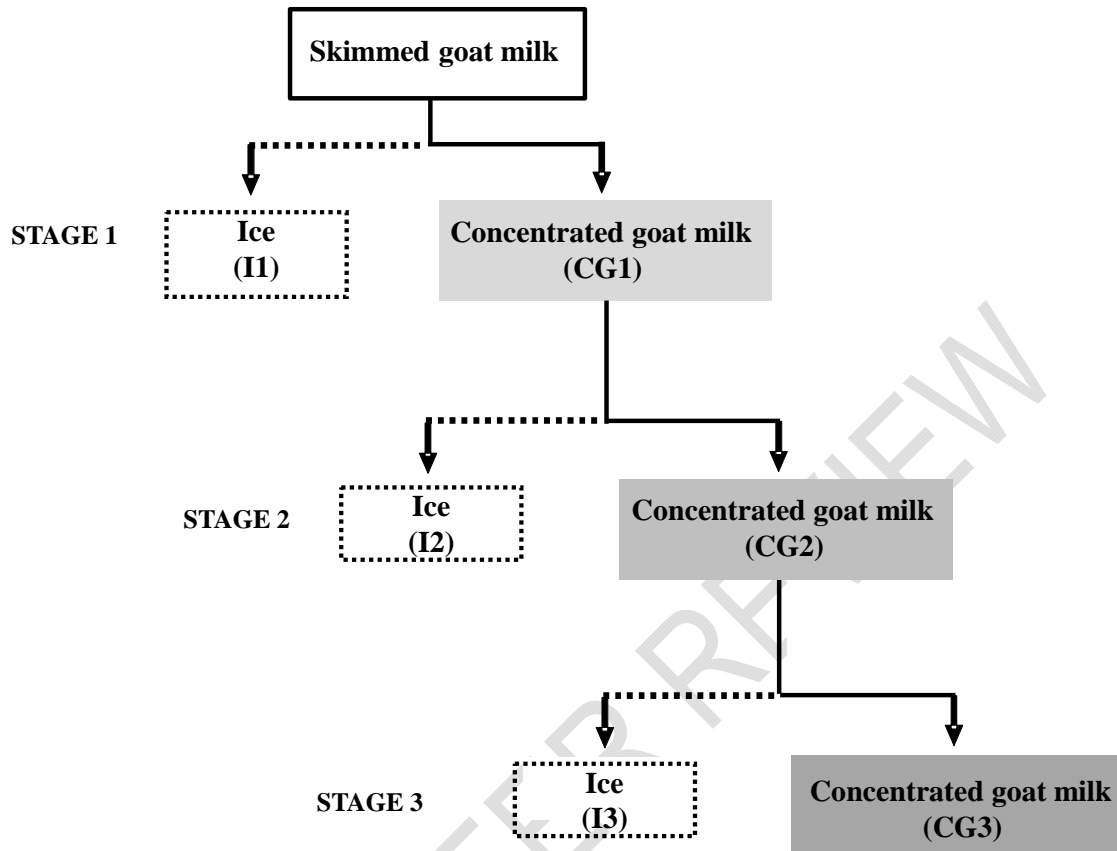


Fig. 1. Diagram of the skim goat milk block freeze concentration process.

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 realized 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 the compressed air. Before the measurement, all samples were calcined at $520\text{ }^{\circ}\text{C}$, 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.

112 **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

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115 **2.3.2 Phosphorus content**

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

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124 **2.3.3 Sodium and potassium content**

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

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134 **2.4 Freeze concentration parameters**

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136 **2.4.1 Concentration factor**

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

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$$142 \quad CF (\%) = \frac{MC_n}{MC_0} \times 100 \quad (1)$$

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144 where MC_n is the mineral (mg kg⁻¹) content of the concentrated goat milk from each freeze
 145 concentration stage and MC₀ is the mineral (mg kg⁻¹) content of the initial skim goat milk.

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147 **2.4.2 Process efficiency**

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

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$$153 \quad eff (\%) = \frac{MC_n - MC_i}{MC_n} \times 100 \quad (2)$$

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155 **2.5 Statistical analysis**

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

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162 3. RESULTS AND DISCUSSION – LEAVE THE SPACE BEFORE MENTIONING 163 OF THE UNITS

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

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185 **Table 2. Mineral contents performance of skim goat milk, concentrated (CG1, CG2,
 186 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 ^{dB}	6.97 ± 0.03 ^{dB}
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 ± 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.

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

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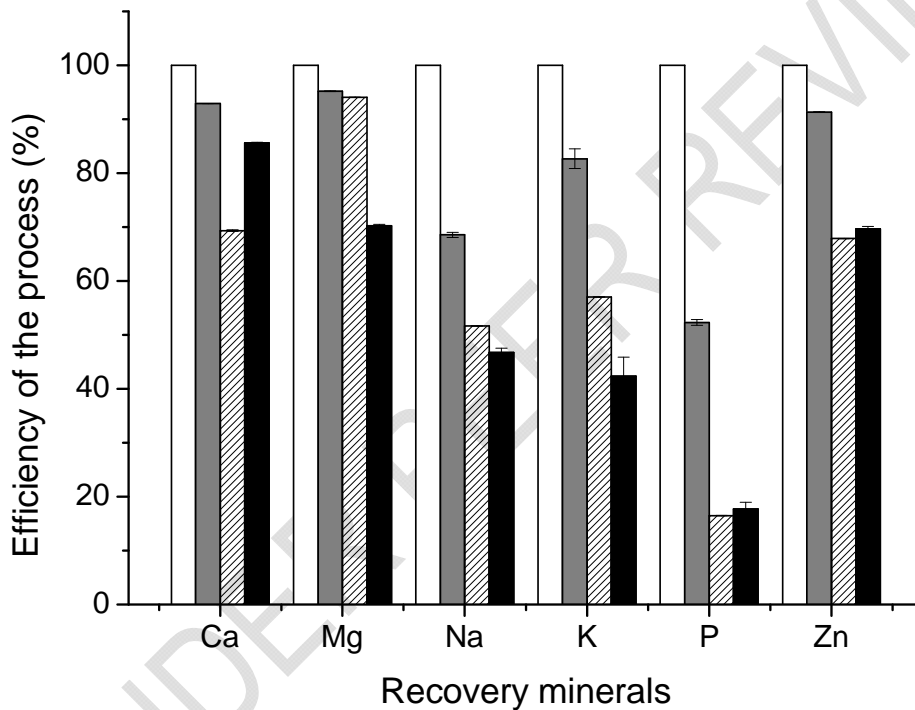
196 Minerals content of Ca, P, K, Na and Mg were higher than those founded by Balde and Aider
 197 [20] during the block freeze concentration of skim cow milk. This performance could be

198 related to the fact the goat milk present some mineral contents, such as Ca, P, K and Mg, in
199 larger amounts than cow milk [5-7].

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201 Regarding mineral efficiency concentration (Fig. 2), overall notable values were achieved.
202 However, the best value was obtained at concentration of Mg with an efficiency of
203 approximately 95 % in the first stage and around 70% at the third stage. The lowest
204 efficiency was to P concentration with an efficiency of 52%, 16%, and 17% at the first,
205 second, and third stages, respectively. In general, the highest process efficiencies were
206 recorded at the end of the first freeze concentration stages. These results indicate that more
207 minerals were entrapped in the ice fraction at the final stages of freeze concentration
208 process (I2 and I3). This performance was also stated by Aider, de Halleux, and Melnikova
209 [32] for the freeze concentration of skim acidic milk.

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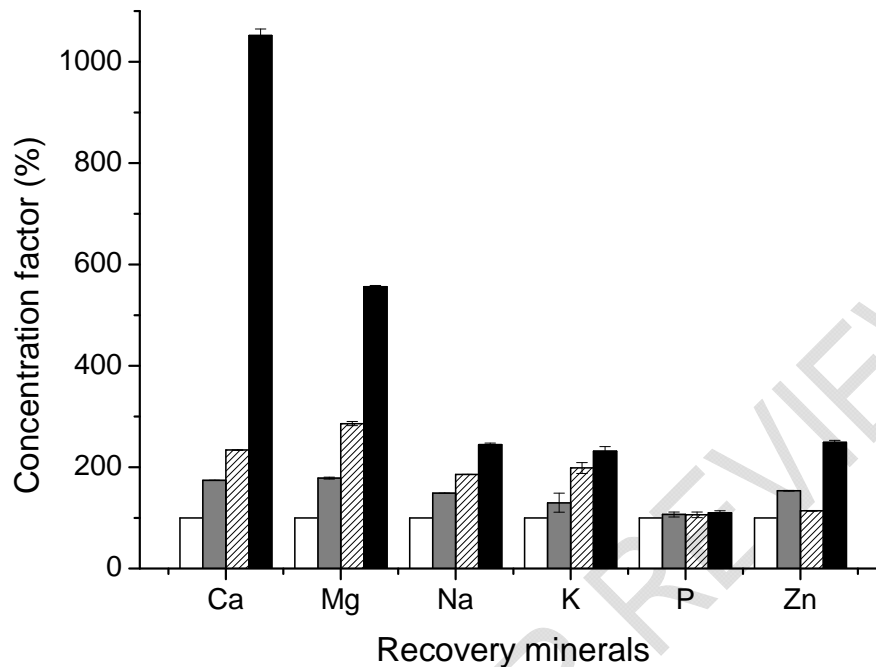
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Fig.2. Performance of freeze concentration process on the efficiency (eff) of goat milk minerals concentration as a function of freeze concentration stages (□ initial skim goat milk, ■ stage 1, ▨ stage 2, and ■ stage 3).

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



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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).

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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 highlight that contents main elements contents of skim goat milk are noted 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 usually used by dairy industries, in order to produce nutritive products with high mineral contents without mineral supplementation, which positively affects the economic and the nutritive value of milk products.

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4. CONCLUSION

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The mineral content of skim goat milk was successfully freeze concentrated by applying the block freeze concentration. As the freeze concentration stages increase, 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 phosphate 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. At least of results obtained on the freeze concentration performance of mineral content, we concluded that the concentrated from the first stage was the best. This is because in this stage was reached higher efficiencies results.

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250 **COMPETING INTERESTS**

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252 Authors have declared that no competing interests exist.

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