Microchemical Journal 129 (2016) 293–296

Contents lists available at ScienceDirect

Microchemical Journal

journal homepage: www.elsevier.com/locate/microc

Decomposition method in semi-closed system with cold finger for evaluation of Ca, K, Na, Mg, Zn and Fe in colostrum silage by F AAS and F AES

Camila Corrêa Pereira [a], Helena Reissig Soares Vitola [b], Alexander Ossanes de Souza [a], Aline Lisbôa Medina [a], Márcia Arocha Gularte [b], Anderson Schwingel Ribeiro [a,*]

[a] Universidade Federal de Pelotas (UFPel), Programa de Pós-Graduação em Química, Laboratório de Metrologia Química (LabMeQui), Capão do Leão, RS, Brazil
[b] Departamento de Ciência e Tecnologia Agroindustrial, Universidade Federal de Pelotas, 96160-000 Capão do Leão, RS, Brazil

ARTICLE INFO

Article history:
Received 3 June 2016
Received in revised form 12 July 2016
Accepted 12 July 2016
Available online 14 July 2016

Keywords:
Cold finger
Silage
Colostrum
Bovine
Minerals
Atomic spectrometry

ABSTRACT

The objective of this study was to use a method of acid decomposition in a semi-closed system with cold finger in colostrum silage samples, for the determination of Ca, Fe, K, Mg, Na and Zn. Colostrum silage samples from five different bovine were studied. Analyte determinations were carried out by flame techniques. In the decomposing system, 5 mL of sample and 5 mL of HNO_3 per 200 °C were used for 2 h, ending with the addition of 1 mL of H_2O_2 for 30 min at 120 °C. Accuracy was evaluated by recovery tests, with results ranging from 83 to 120%, and by the low RSD values (<11.2%). Results differed significantly (95% confidence) between samples with higher levels than those found in milk and this fact reveals that the silage process allows the maintenance of minerals compounds present in colostrum. Furthermore, the values for the elements found in the colostrum silage are close to the values found in the literature for colostrum.

1. Introduction

Colostrum, the first secretion of the mammary gland after birth, plays an important role in the development of newborn calves. In evolution of lactation, this secretion is gradually replaced by milk [1–3]. Studies [4,5] show that colostrum is a source of not only antibodies and nutrients, but also a great number of bioactive compounds such as insulin, insulin-like growth factors (IGFs), growth hormone, prolactin, thyroid hormones, cortisol as well as other compounds including proteins, minerals, vitamins and lipids. According to Foley and Otterrby [6], there is a reduction in the nutritional quality of this secretion soon after birth.

As an alternative to the use of surplus colostrum, producers have used colostrum silage to feed newborn animals [6]. Colostrum silage is the anaerobic fermentation of colostrum by lactic acid bacteria, present in natural microbial secretion, which are mainly *Lactobacillus* [7]. This process enables a better product conservation through lactose conversion into lactic acid, resulting in a pH reduction that directly influences the biological characteristics, preventing the growth and development of pathogenic and spoilage microorganisms [8]. Due to the advantages that colostrum and silage present, they became potential sources of food for human beings, since they have been studied and marketed as food supplements in many countries [9–11].

As regards to the content of minerals in milk and colostrum, the most common macro-elements are Ca, P, Na, K, Mg and Cl, whereas among the trace elements Fe, Cu, Mn, Zn, Co and recently Se have also been studied [12–15]. For mineral determination in these samples, analyses are usually carried out through flame atomic absorption spectrometry (F AAS) [13], graphite furnace atomic absorption spectrometry (GF AAS) [16,17], thermospray flame furnace atomic absorption spectrometry (TS FFAAS) [12] or optical emission spectrometry with inductively coupled plasma (ICP OES) [18,19]. Literature has reported mineral determination in dairy products using spectrometric techniques. The sample in this case is digested by acid decomposition, and performed through digester blocks or plates in open systems [13,20] closed systems with microwave oven [14,20] or alkaline solubilization, such as with tetramethylammonium hydroxide [18]. Some of these systems promote increased risk of contamination and loss of analytes by volatilization. Additionally, a large volume of acid is required, and the microwave equipment has high cost for most laboratories. To our knowledge, there are no reports about mineral content in colostrum silage.

Since colostrum and colostrum silage possess the possibility of being used in food as well as in animal feeding, it is evident the need to develop an efficient sample preparation method for further elemental quantification. Therefore, the aim of this study was to evaluate an adequate preparation procedure for colostrum silage sample using a semi-closed acid decomposition system with cold finger for the determination of Ca, Fe, K, Mg, Na and Zn by flame techniques (F AAS and F AES).

* Corresponding author.
E-mail address: andersonsr@pq.cnpq.br (A.S. Ribeiro).

http://dx.doi.org/10.1016/j.microc.2016.07.012
0026-265X/

Table 1
Instrumental parameters for Fe, K, Mg, Na and Zn determination by F AAS.

Analyte	Wavelength (nm)	Operational mode	Spectral band pass (nm)	Lamp current (mA)
Fe	248.33	Absorption	1.8	30
K	766.49	Emission	2.7	–
Mg	285.21	Absorption	2.7	6
Na	589.00	Emission	1.8	–
Zn	213.86	Absorption	2.7	15

2. Material and methods

2.1. Instrumentation

The determination of Fe, K, Mg, Na and Zn was carried out using a flame atomic absorption spectrometer (F AAS) (AAnalyst 200, Perkin Elmer, Shelton, CT, EUA) which allows operate in emission mode, equipped with hollow cathode lamps for every determined analyte (Lumina, Perkin Elmer, Shelton, CT, EUA) and a deuterium arc background correction lamp. The flame consisted of a mixture of air/acetylene 99.7% (Linde, São Paulo, Brazil) as oxidant/fuel gas, respectively. Instrumental parameters used for each analyte are presented in Table 1.

For Ca determination, a flame atomic emission spectrometer was used (F AES) (B462, Micronal, São Paulo, SP, Brazil). Liquefied petroleum gas (LPG) was used as fuel and compressed air as oxidant gas. Analyzes were performed under the following conditions: sample volume of 5 mL min^{-1} and air 9 L min^{-1} at a pressure of 1 kgf cm^{-2}.

For the acid decomposition sample, a heated digester block was used (MA-4025, Marconi, Piracicaba, SP, Brazil). In the digestion tubes, a reflux system was coupled with a PTFE cap with a side slot for pressure relief, with internal circulation of water at a temperature of 15 °C, cooled by a thermostatic bath (Q-214M2, Quimis, São Paulo, SP, Brazil). This reflux system was developed by our research group and is described by Oreste et al. [21].

2.2. Reagents

Analytical reagent grade materials were used and solutions were prepared using ultrapure water, which was obtained employing a Direct-Q 3 Water Purification System (Millipore Corporation, USA) with a resistivity of 18.3 MΩ cm at 25 °C. The solutions of all analytes were prepared in acid medium by appropriate dilutions of a stock solution containing 1000 mg L^{-1} (Merck, Darmstadt, Germany) in deionized water.

For sample preparation, nitric acid 65% (w/w) (Vetec, Rio de Janeiro, RJ, Brazil) was purified by doubly sub-boiling distillation in a quartz system MA-075 (Marconi, Piracicaba, SP, Brazil). Additionally, this step also used hydrogen peroxide 30% (Merck, Darmstadt, Germany). Cesium chloride 1% w/v and lanthanum chloride 10% w/v (Fluka, Buchs, Germany) were used as ionization suppressor and formation of oxides respectively, for the determination of Mg by F AAS and Ca by F AES. All material used were washed with water and detergent, dried in ambient temperature and soaked in 10% (v/v) HNO_3 for at least 48 h, and then rinsed with deionized water and dried.

2.3. Samples

Five colostrum silage samples were obtained from the Farmers Training Center of Canguçu (*Centro de Treinamento de Agricultores de Canguçu, CETAC*) and numbered as 1, 2, 3, 4 and 5 (from 5 different animals). The samples were stored under refrigeration at 4 °C until analysis.

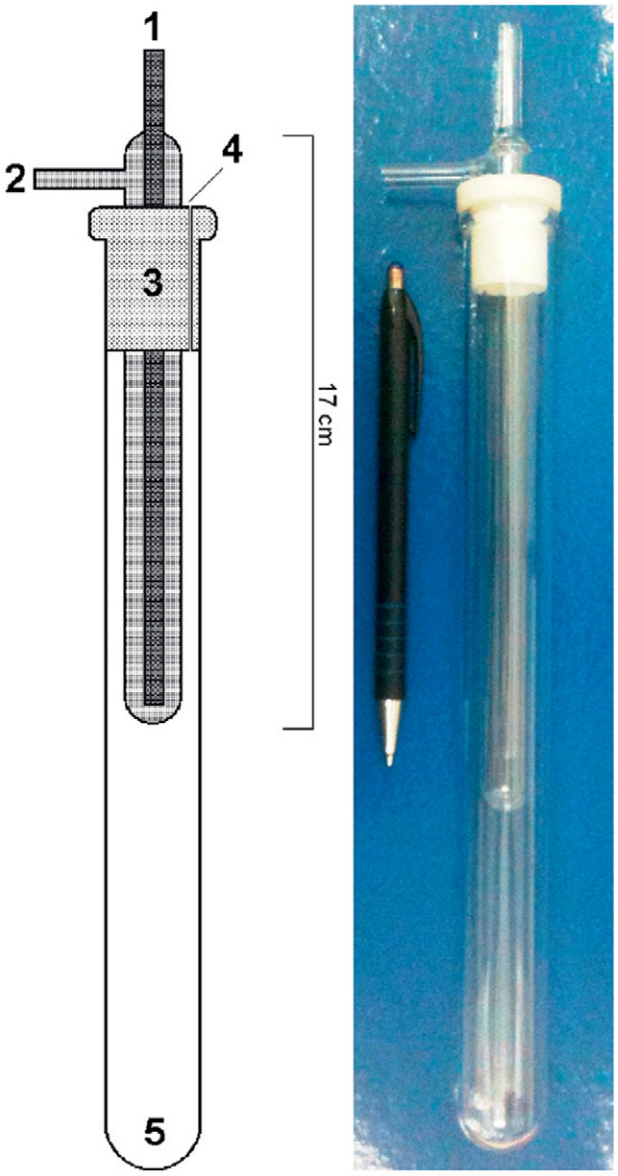

Fig. 1. Schematic diagram of cold finger coupled to glass digester tube. 1: water inlet; 2: water outlet; 3: end cap of PTFE; 4: gas outlet; 5: reaction flask. B) Picture of cold finger system.

2.4. Sample preparation

Initially, 5 mL of colostrum silage was weighed directly in the decomposition tubes, followed by the addition of 5 mL of HNO_3 and glass beads of approximately 4.0 mm to minimize solution outbreaks during heating. Afterwards, the reflux system was coupled to the tubes and brought to the digester heating block, where it remained for 2 h at 200 °C. Thereupon, the solutions were cooled at room temperature for further adding of 1 mL of H_2O_2, and forwarded again to the digester block for 30 min at 120 °C. At the end of the procedure, the samples were transferred into polyethylene bottles, adjusting the volume to 15 mL with deionized water. All samples were prepared in triplicate and analytical blanks were prepared by the same procedure. For accuracy verification, a recovery test was carried out in samples 1 and 2, after the addition of 150, 225 and 300 mg L^{-1} of Ca, 1.5, 3.0 and 6.0 mg L^{-1} of Fe, 300, 750 and 1500 mg L^{-1} of K, 45, 95, 135 mg L^{-1} of Mg, 150, 300 and 450 mg L^{-1} of Na, 3.0, 7.5, 15.0 mg L^{-1} of Zn.