Determination of K, Mg and Na in milk powder using a new analytical approach

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Abstract

MIC was applied for sample preparation of milk powder for further K, Mg and Na determination by LEP-OES.

Introduction

Some minerals, such as Na, K and Mg, are considered essential to life. These elements are responsible for the maintenance of osmotic pressure, transmission of nerve impulses and contraction of muscles besides being essential constituents of skeletal structures and many enzymes, vitamins and hormones (Campbell, 2011). The determination of these elements in milk powder is important for evaluating their nutritional and/or toxicological properties as well as controlling the quality of the product. That is why the importance of knowing about the concentration of these essential mineral elements in food is widely recognized. Liquid Electrode Plasma Optical Emission (LEP-OES) is a novel elemental analysis method and an alternative to Na, K and Mg determination. It consists of using high-voltage direct current (DC) pulse power to generate a microplasma inside of a sample solution (Banno, 2009). LEP-OES has some advantages when compared to techniques commonly used, such as low cost, low energy consumption and portability, since it requires no plasma gas and no high power source. Nevertheless, it is very susceptible to the presence of high amounts of dissolved carbon and/or high acid concentrations in solution. Therefore, a suitable sample preparation is crucial to the success of the analysis, and the microwave-induced combustion (MIC) has been proposed as an alternative for complete digestion of organic matrices using diluted acids (Pereira, 2013). In this study, K, Mg and Na were determined by LEP-OES after digestion by MIC.

Results and discussions

Whole and skim powder milk were purchased in markets from Pelotas/RS/Brazil. Samples were homogenized, dried at 65 ºC for 4 h and digested by MIC using 500mg of sample mass and 6 ml of HNO_3 4 mol l^{-1}, as the absorbing solution for analytes. After, a sample was chosen randomly for the optimization of the LEP-OES measurement conditions. According to Banno (2009), the sensitivity for each element in LEP-OES depends on the number of pulses (NP), pulse width (PW), period between the pulses (PBP) and voltage applied (VA). Thus, with the exception of the PBP, these parameters were investigated using a central composite design (2^3 with 3 central points and 6 axial points, totaling 17 experiments). The following measurement conditions were evaluated: NP (1 to 101), PW (0.2 to 10.0 ms) and PBP (0.2 to 60.0 ms). These experiments were performed using the MH-5000 ultracompact elemental analyzer (Micro Emission Ltd., Japan) and a quartz chip (LepiCuve-C cuvette). The analytical response recorded was emission intensity, and the experimental data were processed using STATISTICA 7.0 software (StatSoft, Inc., Tulsa, OK, EUA).The results of the factorial design for K (766.491 nm) demonstrated that, among the measurement conditions studied, only the PBP was significant, having an overall positive effect. Moreover, the interaction of the PBP and the PW was also significant. On the other hand, no significant variable occurs for Na (588.995 nm) and Mg (518.362 nm). Thus, taking into account the optimized parameters and the background for all signals, the most appropriate measurement conditions were selected (PN = 20; PW = 7 ms and PBP = 50 ms). Afterward, the VA (600 to 900 V) was evaluated, and 700 V was selected due the lower background signal. Recovery tests were performed in order to verify the accuracy of the proposed method, and the recoveries for K, Mg and Na, ranged from 94 to 107%. Furthermore, the limits of detection (LODs) were 173 mg kg^{-1}, 175 mg kg^{-1} and 25.0mg kg^{-1} for K, Mg and Na, respectively. Thus, the proposed method was applied for whole milk powder (K: 6932 ± 512 mg kg^{-1};Mg: 1580 ± 138 mg kg^{-1} and Na: 4639 ± 371 mg kg^{-1}) and skim milk powder (K: 7657 ± 611 mg kg^{-1}; Mg: 1502 ± 124 mg kg^{-1} and Na: 5277 ± 493). The relative standard deviations (RSDs) were up to 9%, which may be considered suitable for this technique once that small sample volume (60 μl) is used.

Conclusion

Based on obtained results, MIC provides suitable solutions for further determination by LEP-OES. Moreover, the results showed accuracy, precision and suitable LODs. Finally, this new analytical approach can be successfully applied for milk powder analysis contributing for food quality control, and it is able to minimize the environmental damage since it uses small volumes of reagents.

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