Feasibility of Single Reaction Chamber for Margarine Digestion and Subsequent Determination of Ni by ICP-MS

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Introduction

The quantitative determination of Ni in margarine is very important considering the toxicity of this element, and also depending on concentration, it might affect the quality of this product.[1] The occurrence of Ni in margarine is generally from the production process, which usually employs this element as a catalyst.[2,3] Thus, considering the importance of Ni control, the high fat content of the margarine and the few methods available for the metal determination in these products, it is necessary the development of new analytical methods suitable for the determination of Ni in margarine.

In the present work, a Single Reaction Chamber system (SRC) was proposed for margarine digestion and further Ni determination by inductively coupled plasma mass spectrometry (ICP-MS). This digestion system combines high pressure and temperature with the efficient distribution of microwave radiation into the reaction chamber unit. The efficiency of digestion was evaluated by determination of residual carbon content (RCC).

Results and Discussion

Margarine samples, without salt, were purchased at the local market (Pelotas - RS – Brazil), and they were decomposed in a SRC (UltraWave^{TM} system, Milestone, Italy) equipped with 5 quartz digestion vessels. In this method, the sample mass (500 to 800 mg) and 10 mL of HNO_3 solution (2, 4, 6, 8, 10 or 14.4 mol L^{-1}) were evaluated. Digestion vessels were introduced into a reaction chamber containing 2 mL of H_2SO_4, 5 mL of H_2O_2 and 120 mL of water. The reaction chamber unit was pressurized with 30 bar of Ar, and the applied heating program was: *i)* 10 min of ramp; *ii)* 150 ºC/10 min; *iii)* 10 min of ramp; and *iv)* 250 ºC/15 min. Pressure and microwave power were limited at 160 bar and 1500 W, respectively. RCC was determined in the final digests by inductively coupled optical emission spectrometry (ICP OES, Spectro Ciros CCD, Spectro Analytical Instruments, Germany), and Ni was determined by ICP-MS (Elan DRC II, PerkinElmer, Canada). The accuracy of the proposed method was evaluated using a certified reference material (CRM) BCR 414 (plankton), which was decomposed under the same conditions of the margarine samples. Using a 2 mol L^{-1} HNO_3 solution, the digestion of samples was not effective (RCC higher than 2700 mg L^{-1}) and digests with a dark colored aspect were obtained. However, using 4 mol L^{-1} HNO_3 or higher concentrations, it was possible to digest up to 800 mg of margarine and, as expected, RCCs decreased according to the increased nitric acid concentration. Nickel was determined in final digests, and no significant differences (ANOVA, $p>0.05$) were observed in the Ni concentration using diluted HNO_3 solutions. However, in order to avoid deposits of carbon in the interface of ICP-MS equipment, 8 mol L^{-1} HNO_3 was selected as a suitable digestion solution (RCC: 254 ± 24 mg L^{-1}) to decompose up to 800 mg of margarine in almost 45 min. In these selected conditions, the result for Ni (18067 ± 1029 µg kg^{-1}) in CRM BCR 414 was in agreement (96%) with certified value (18800 ± 800 µg kg^{-1}), showing a good accuracy of the proposed method. Using this method, the limit of detection for Ni was 11.8 µg kg^{-1}. The Ni concentration in the margarine sample was 130.2 ± 4.6 µg kg^{-1}, which is lower than the maximum limit established by the Brazilian Government for this type of food (4 mg kg^{-1}).[4] Moreover, it is important to emphasize that this study is in process and this method should be applied for margarine samples from different manufacturers.

Conclusions

The Single Reaction Chamber was suitable to decompose up to 800 mg of margarine using 10 mL of 8 mol L^{-1} HNO_3 and to further Ni determination by ICP-MS. Moreover, the use of SRC with diluted HNO_3 allows obtaining digests with suitable RCC for ICP-MS analysis. The Ni concentration in the evaluated sample was lower than the maximum limit established by Brazilian legislation.

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