Determination of Ni in Chocolate by ICP-MS after Microwave-assisted Digestion Combined with Ultraviolet Radiation

Dirce Tainá T. de Oliveira[1] (IC), Diogo L. Novo[1] (PG), Sandra M. Cruz[2] (PG), Fabio A. Duarte[2] (PQ), Márcia F. Mesko[1] (PQ), Carla A. Hartwig[1] (PG)* (*carlahartwig@yahoo.com.br)

[1]*Centro de Ciências Químicas, Farmacêuticas e de Alimentos, Universidade Federal de Pelotas, Pelotas – RS*
[2]*Departamento de Química, Universidade Federal de Santa Maria, Santa Maria - RS*

Keywords: *Chocolate, Nickel, MW-UV.*

Introduction

Chocolate is a widely consumed food, mainly in the form of bars. However, this type of sweet can have toxic metals in its composition, which can change the quality of the product and cause damage to health such as allergies and other problems caused by ingestion of Ni.[1,2] Thus, the estimated amount of Ni to ingest daily for balanced diet is 50 mg.[2] The main sources of Ni in chocolates are the raw materials and processes of manufacturing and storage.[1]

In this study, the combination of ultraviolet radiation and microwave-assisted digestion (MW-UV) was evaluated for digestion of chocolate samples using diluted nitric acid as digesting solution. The Ni concentration was determined by inductively coupled plasma mass spectrometry (ICP-MS), and the efficiency of digestion was evaluated through the residual carbon content (RCC) in the final solutions, which was determined by inductively coupled optical emission spectrometry (ICP-OES).

Results and Discussion

The samples purchased in markets in different regions of Brazil, were decomposed (500 mg) by MW-UV in a microwave oven (Multiwave 3000^{TM}, Anton Paar) equipped with eight quartz vessels (80 ml, 80 bar and 280 °C). One sample was arbitrarily selected for the optimization procedure, when were evaluated 2, 4, 7 or 14.4 mol l^{-1} HNO$_3$ (10 ml) as digestion solution. The UV radiation was conducted by inserting UV lamps into the vessels. The microwave heating program used was: 20 min of ramp, 550 W for 10 min, 10 min of ramp, 900 W for 20 min and 0 W for 15 min. The RCC was determined in each evaluated condition, and the best condition was applied to digest other samples. The certified reference material (CRM) BCR 414 (plankton) was decomposed under the same conditions as the sample to evaluate the accuracy of the method. Using 2 mol l^{-1} HNO$_3$ as the digestion solution, the RCC in the final solutions was higher than 7000 mg l^{-1}, which can cause problems in the ICP-MS determination. On the other hand, the use of HNO$_3$ in concentration equal to or higher than 4 mol

l^{-1} showed good digestion efficiency, with RCC lower than 100 mg l^{-1} in all final solutions.

Therefore, considering the efficiency of digestion and the possibility of using diluted solution, 4 mol l^{-1} HNO$_3$ was selected as the digestion solution for chocolate and subsequent Ni determination by ICP-MS. In this condition, the limit of detection (LOD) achieved for Ni was 56.7 µg kg^{-1}. The accuracy of the method was verified by good agreement (97%) between the Ni concentration obtained for CRM (18254 ± 1007 µg kg^{-1}) and its certified value (18800 ± 800 µg kg^{-1}). Nickel was determined in six samples of milk chocolate (MC) and white chocolate (WC) from different manufacturers. The results are shown in Table 1.

Table 1. Nickel concentration determined by ICP-MS in chocolate after MW-UV digestion (n = 3).

Sample	Ni (µg kg^{-1})	Sample	Ni (µg kg^{-1})
MC 1	559.4 ± 31.9	WC 1	104.8 ± 7.5
MC 2	537.6 ± 38.7	WC 2	< 56.7
MC 3	380.9 ± 25.7	WC 3	< 56.7

As can be observed, the highest Ni concentrations were obtained for milk chocolate samples, so it can be associated with the presence of cacao in these samples.[1]

Conclusions

The use of MW-UV and ICP-MS was suitable for Ni determination in chocolate samples. Moreover, the decomposition allowed the use of diluted HNO$_3$ as a digestion solution and suitable LOD was obtained.

This study is in process and the proposed method will be applied to other types of chocolate and will determine other elements.

Acknowledgement

[1]Jalbani, N; et al. J. Anal. Environ. Chem. 10, **2009**, 48.
[2]Carrapatoso, I; et al. Revista Portuguesa de Imunoalergologia, XII, **2004**, 261.