Evaluation of MIC for Sample Preparation of Whole Egg Powder and its Fractions with further Br and I Determination by ICP-MS

Gilberto da S. Coelho Junior[1] (IC), Thais Mazzetti[1] (IC), Isis G. Toralles[1] (PG), Sandra M. da Cruz[2] (PG), Clarissa M. Moreira[2] (PQ), Márcia F. Mesko[1] (PQ)* – (marcia.mesko@pq.cnpq.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: *Bromine, Iodine, Egg, MIC, ICP-MS.*

Introduction

The importance of iodine for human nutrition is well known mainly because it is necessary in the synthesis of thyroid hormones. On the other hand, bromine can also be classified as a trace element with possible essential function; however it is not very well described in the literature.[1] Therefore, knowledge about the Br and I concentration in foods is very important in view of controlling the intake of these elements. In this sense, it is important to emphasize that eggs are widely consumed in the world and there is little information about halogens concentration in this type of food, especially for Br. Thus, the development of suitable analytical methods to quantify Br and I in eggs is a challenge, mainly considering the complex sample matrix, which presents high content of fat and protein. In this sense, microwave-induced combustion (MIC) associated with diluted alkaline solutions has been used as an alternative to decompose organic samples for subsequent determination of halogens.[2] Furthermore, especially for Br and I determination in low concentrations inductively coupled plasma mass spectrometry (ICP-MS) has been considered a very convenient technique.[3] In this work, whole egg powder and its fractions were decomposed by MIC for subsequent Br and I determination by ICP-MS.

Results and discussion

Whole egg powder and its fractions (white and yolk) were purchased in markets from different regions of Brazil. For sample decomposition, polyethylene (PE) wrappers containing sample (100 to 450 mg) and 100 µl of NH_4NO_3 (6 mol l^{-1}) were prepared and placed on the base of a quartz holder, with a small piece of filter paper containing 50 µl of 6 mol l^{-1} NH_4NO_3. Then, the samples and holders were transferred to quartz vessels containing 6 ml of absorbing solution (H_2O or 10 to 75 mmol l^{-1} NH_4OH). The vessels were closed, pressurized (20 bar of O_2), fixed to the rotor and placed inside of the microwave oven (Multiwave 3000™, Anton Paar). The microwave heating program applied was as follows: *i)* 1400 W/50 s; *ii)* 0 W/1 min; *iii)* 1400 W/5 min and *iv)* 0 W/20 min. The resultant solutions were analyzed by ICP-MS. By using the MIC method, it was possible to digest up to 350 mg of samples, in view that they present high content of fat and protein. As shown in Fig. 1, using water or 10 mmol l^{-1} NH_4OH, recoveries for Br and I were higher than 93% for both analytes. However, relative standard deviations (RSDs) were between 9 and 12%. On the other hand, when 50 mmol l^{-1} NH_4OH or higher concentrations were used as absorbing solution, recoveries were between 98 and 102% for both analytes, and RSDs were up to 6%. Moreover, it is important to mention that the pH measurements in the digests using water or 10 mmol l^{-1} NH_4OH were around 3, which is not suitable for Br and I stability in solution. However, using 50 mmol l^{-1} NH_4OH or superior concentration, the pH ranged between 7 and 8, and, in this case, 50 mmol l^{-1} NH_4OH was considered suitable for Br and I absorption.

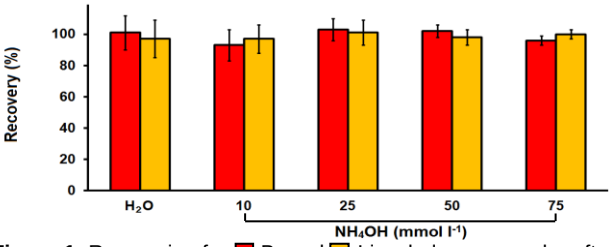

Figure 1. Recoveries for ■ Br and ■ I in whole egg powder after MIC/ ICP-MS (n=3).

In order to evaluate the accuracy of the proposed method, a RM NIST 8435 (whole milk powder) was decomposed in the same conditions of the samples. The agreements between the obtained results (Br: 19.1 ± 0.7 µg g^{-1} and I: 2.37 ± 0.06 µg g^{-1}) and reference values (Br: 20.0 ± 10.0 µg g^{-1} and I: 2.30 ± 0.40 µg g^{-1}) were better than 95% for both analytes. Moreover, the limits of detection using the MIC method were 0.039 µg g^{-1} for Br and 0.015 µg g^{-1} for I. Thus, egg fractions were also decomposed by the MIC method using 50 mmol l^{-1} NH_4OH solution. Bromine and I were obtained in trace concentrations in whole egg powder (Br: 5.29 µg g^{-1} and I: 0.66 µg g^{-1}) and its fractions: white (Br: 16 µg g^{-1} and I: 0.15 µg g^{-1}) and yolk (Br: 2.53 µg g^{-1} and I: 1.47 µg g^{-1}).

Conclusion

The MIC method was efficient for decomposition of whole egg powder and its fractions, with quantitative recoveries for Br and I, using NH_4OH 50 mmol l^{-1} as absorbing solution. Furthermore, the MIC method presented a high throughput (16 samples/h) and suitable accuracy.

Acknowledgments

Capes, CNPq, Fapergs, LAQIA/UFSM and UFPel.

[1] Romarís-Hortas, V. et al. *Talanta*. **2009**, *79*, 947.
[2] Hartwig, et al. *Analytical Methods*. **2014**, *6*, 7540.
[3] Knapp et al. *Fresenius J. Anal. Chem.* **1998**, 362, 508.