

Determination of Bromine and Iodine in Rice by ICP-MS after Digestion by Microwave-induced Combustion

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Introduction

Rice is one of the most important cereals and it is consumed by more than half of the world's population. The types of rice available for consumption (brown, parboiled or white) are produced in diverse forms and contain variable amounts of nutrients. Among these nutrients, although I shows important biological functions, when ingested in high concentration, can result in health implications. On the other hand, Br did not present a knowledge function in the human body.^{1,2} Thus, considering the importance of determining these elements in rice, the development of suitable analytical methods is of a great concern, considering that halogens determination is susceptible to losses by volatilization during the sample preparation step. In this study, microwave-induced combustion (MIC) was used for preparation of rice samples, and Br and I were determined in final digests by inductively coupled plasma mass spectrometry (ICP-MS).

Results and Discussion

Samples of white, parboiled and brown rice, purchased at a local market (Pelotas – RS), were ground and dried in an oven at 60 °C for 4 h. For decomposition by MIC, a microwave oven (Multiwave 3000™, Anton Paar) equipped with up to 8 quartz vessels (80 mL, 280 °C and 80 bar) was used. The samples of rice (500 to 1100 mg) were wrapped in a polyethylene film. A quartz holder, containing a filter paper with 50 µL of 6 mol L⁻¹ NH₄NO₃, and the sample were inserted into a quartz vessel previously charged with 6 mL of absorbing solution (ultrapure water or 25 to 100 mmol L⁻¹ NH₄OH). The vessels were closed, pressurized with 20 bar of O₂ and irradiated with microwaves (1400 W/5 min; 0 W/20 min).³ Bromine and I were determined by ICP-MS (Elan DRC II, PerkinElmer, Canada). The accuracy of the proposed method was evaluated by analyses of certified reference material (CRM) NIST 8435, submitted to the same conditions of the samples, and also through recovery tests. By using the MIC method, it was possible to digest up to 1000 mg of rice. Thus, based on recovery tests, it was possible to observe that using water or 25 mmol L⁻¹ NH₄OH recoveries for Br and I ranged from 88 to 98% with relative standards deviations (RSDs) up to 15%. However, with 50 mmol L⁻¹ NH₄OH, or higher

concentrations, recoveries of Br and I ranged from 95 to 102%, with RSDs up to 7%. Therefore, 50 mmol L⁻¹ NH₄OH was selected as absorbing solution for Br and I in rice. Using the selected conditions, it was possible to obtain agreements of 97 and 102% for Br and I, respectively, with the informed values in the CRM. The limits of detection attained by the proposed method were 0.009 µg g⁻¹ for Br and 0.005 µg g⁻¹ for I. The results obtained for the analysis of the rice samples are shown in Table 1.

Table 1. Determination of Br and I by ICP-MS in rice after decomposition by MIC (n=3).

Rice	Concentration (µg g ⁻¹)	
	Br	I
White	0.398 ± 0.004	0.021 ± 0.001
Parboiled	0.266 ± 0.018	0.015 ± 0.001
Brown	0.454 ± 0.031	0.021 ± 0.001

As it is possible to observe in Table 1, Br concentration is higher in brown rice, which could be related to the treatment that this type of rice is submitted and can suggest that Br probably can be a result of some anthropogenic activity. For I, this behavior was not observed (concentrations of I in white and brown rice were similar), probably because this element is naturally in rice. However, parboiled rice shows a lower concentration of Br and I in comparison with other types of rice, and it could be related to the pre-cooking process. In addition, it is important to emphasize that Br and I in the evaluated samples are in concentrations below the recommended daily intake limits (8 mg/day for Br; 150 µg/day for I)^{1,2} considering the ingestion of a portion of 50 g of rice.

Conclusion

Based on the obtained results, it was found that the MIC associated with ICP-MS is suitable to the Br and I determination in rice. By using the proposed method, it was possible to observe differences in the analytes concentration in white, parboiled and brown rice, especially for Br. In addition, it is important to mention that this study is in process and will be applied for the rice from other origins.

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¹World Health Organization, Geneva, 2007.

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