Microwave-assisted wet digestion of yerba-mate with diluted acid in closed vessel for As, Cd and Pb determination by ICP-MS

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Abstract

The use of diluted HNO_3 was evaluated for the digestion of yerba-mate for further As, Cd and Pb determination by ICP-MS.

Introduction

Yerba-mate (*Ilex paraguariensis*) is a plant highly consumed in countries from South America, such as Argentina, Brazil and Uruguay. This herb is mainly consumed as an infusion and it is related to many therapeutic properties. The mineral composition of this plant may include macronutrients, micronutrients and trace elements, including some toxic contaminants, such as As, Cd, Cr and Pb. These toxic elements are normally present at low concentrations (μg g^{-1} or less), requiring the use of sensitive techniques for reaching specific regulations. Additionally, the sample digestion procedure is normally time-consuming and require the use of concentrated mineral acids.[1] Alternatively, diluted reagents can be used without commit digestion efficiency. As an alternative, microwave-assisted wet acid digestion (MAWD) method has been used with diluted HNO_3 and O_2 pressure in closed vessel. This method uses O_2 to oxidize NO to NO_2, leading to *in situ* reaction for HNO_3 regeneration.[2] In this sense, digestion using diluted HNO_3 was evaluated for decomposition of up to 1 g of yerba-mate. For comparison, a digestion procedure using concentrated HNO_3 without O_2 was also evaluated.

Results and Discussion

Analytes were determined by ICP-MS (PerkinElmer-SCIEX, Model Elan DRC II, Canada) equipped with a concentric nebulizer (Meinhard Associates, USA), and a cyclonic spray chamber (Glass Expansion). Microwave-assisted digestion using diluted (4 and 7 mol L^{-1}) HNO_3 was evaluated for digesting 500 up to 1000 mg of yerba-mate. Quartz vessels were pressurized with 15 bar of O_2. The procedure with 14.4 mol L^{-1} HNO_3 was performed with 500 mg of powdered sample (without O_2 pressure). No significant difference (unpaired Student t-test) was observed between results obtained using diluted or concentrated HNO_3. To evaluate digestion efficiency, residual carbon content (RCC, %) and residual acidity were determined in digests. For sample masses up to 1000 mg, digestion was performed with 4 mol L^{-1} HNO_3 and 15 bar of O_2. Results are shown in Fig. 1.

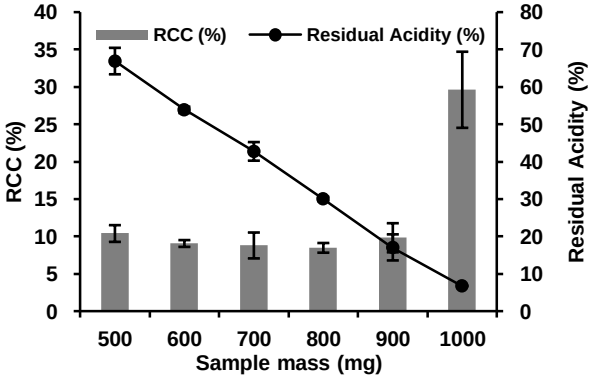

Figure 1. Results for RCC and residual acidity for sample masses from 500 to 1000 mg.

Sample masses up to 900 mg were efficiently digested using diluted HNO_3 when the system was pressurized with O_2. Although digestion of 900 mg presented a relatively low RCC value (9.8%), the same digestion efficiency was not achieved for sample mass of 1000 mg with diluted HNO_3, which resulted in high RCC (30%). In this way, up to 900 mg of sample could be efficiently digested. This condition was suitable for As, Cd and Pb determination by ICP-MS without carbon interferences in the plasma, clogging of introduction system, as well as no need of matrix matching during analysis.

Conclusion

The proposed O_2 pressurized MAWD method allowed the use of diluted HNO_3, being a promising alternative for the digestion of high sample mass of yerba-mate. Even using diluted acids, the obtained RCC values and residual acidity in final digests were low enough for making this procedure suitable for the determination As, Cd and Pb by ICP-MS. Additionally, the relatively high sample mass digested in closed system allowed very low limits of detection for these elements (0.71, 0.13 and 2.1 ng g^{-1} respectively) which are in agreement with legislation requirements.

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