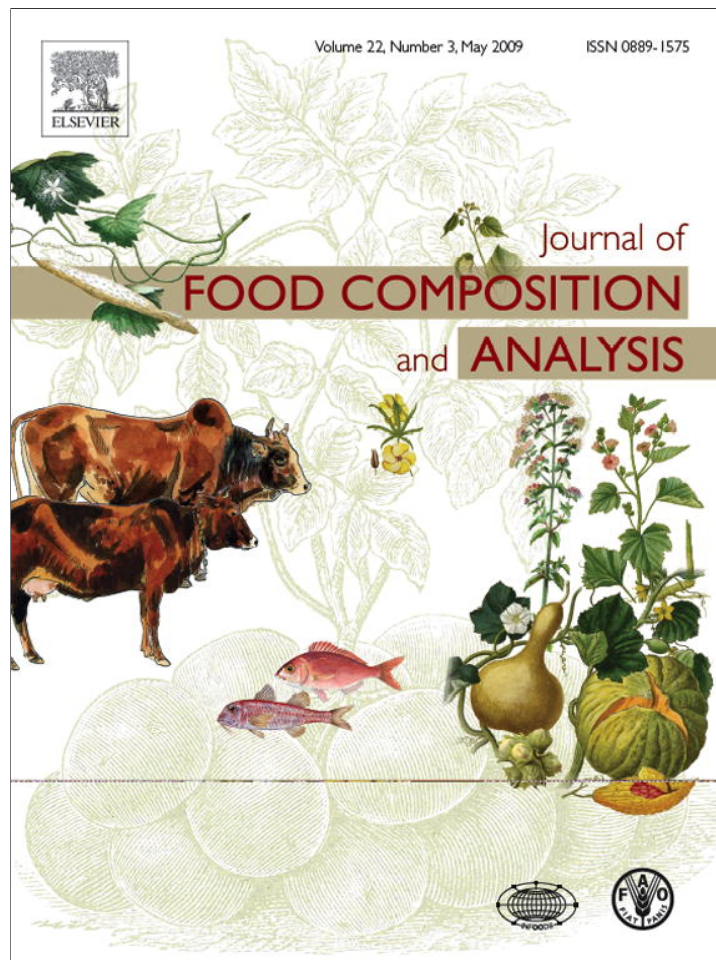


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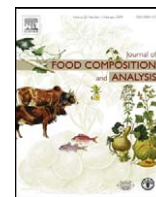
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## Original Article

## Focused-microwave-assisted acid digestion: Evaluation of losses of volatile elements in marine invertebrate samples

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## ABSTRACT

Biological samples can be acid-digested using open or closed reaction vessels. Each one has advantages and limitations related to sample mass, contamination, operational temperature range, and losses of analytes. The latter may be critical when working with thermolabile chemical compounds. In the present work, focused-microwave and cavity-microwave ovens were employed for acid digestion of marine invertebrate samples (oyster, mussel and clam). The aim of this study was to evaluate losses of volatile species containing As, Cd, Pb, Se, and Zn, in both microwave systems, after an acid digestion procedure using nitric and sulfuric concentrated acids. The elemental determinations were performed by inductively coupled plasma optical emission spectrometry (ICP OES) with axially viewed configuration. Using a focused-oven, losses of volatile elements were always higher than 12%, when compared to cavity-microwave oven. High losses were observed for Pb and Cd, while Zn was not affected by volatilization in marine invertebrate samples. Standard reference material (Oyster tissue, NIST SRM 1566b) was used to check the accuracy.

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### 1. Introduction

Microwave-assisted digestion procedures are well established and accepted as a fast alternative for sample preparation. Procedures are carried out using especially designed cavity- or focused-microwave ovens with respective reaction vessels (Kingston and Haswell, 1997; Mermet, 1997). Both can perform properly when applying tailored methods for different types of organic and inorganic samples.

The cavity-microwave oven uses high-pressure closed vessels and is suitable for procedures dealing with organic sample masses typically lower than 0.5 g to avoid excessive pressure build-up and undesirable effects. The microwave radiation is distributed into the cavity, and the rotor traditionally contains 12 reagent vessels.

These procedures are recommended for trace analysis and the materials for production of the reaction vessels were developed for avoiding losses and memory effects even when working at high temperatures and pressures (Kingston and Haswell, 1997; Mermet, 1997; Erickson, 1998).

However, for the digestion of large masses of organic samples, the use of focused-ovens is attractive. The focused-microwave oven employs open vessels, and digestion is carried out at atmospheric pressure. Microwave radiation is focused in the bottom part of the vessel containing the sample and the reagents. The system is advantageous due to its capacity to allow the removal of gases generated during the digestion process. According to the literature, e.g. Mermet, 1997, the term “open” is not appropriate because the vessels are not open during the digestion; instead, there is a purge system to remove the gaseous products formed during the oxidation of the sample.

Despite the more frequent use of cavity-microwave ovens, the application of focused-microwave ovens is also usual and many procedures based on these systems have been developed (Nóbrega et al., 2002). However, in spite of its successful applications, the

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users must pay attention to particularities of this oven. For example, the non-homogeneous distribution of microwave radiation should be mentioned, previously demonstrated for a six-vessel focused-oven (Costa et al., 2001).

Recent applications of focused-ovens emphasized vapor-phase digestions (Araújo et al., 2004; Trevizan et al., 2003), high-pressure asher (Matusiewicz, 1994, 1999), and an alternative strategy based on gradual sample addition to microwave-assisted heated concentrated nitric acid (Nóbrega et al., 2002; Santos et al., 2005). It has already been demonstrated that the alternative procedure, i.e. gradual sample addition to the hot concentrated acid, leads to better efficiency of digestion than the conventional one based on the addition of concentrated acids to the samples. This improved efficiency is obtained using lower amounts of concentrated acids due to more aggressive reaction conditions (Nóbrega et al., 2002; Santos et al., 2005; Trevizan et al., 2007).

Losses of volatile elements when applying focused-ovens may occur as a result of the reaction vessel design. These vessels usually contain an air condenser at the top. Moreover, microwave radiation only interacts with the lower part of the vessel containing the sample (approx. 8 cm, according to Trevizan et al., 2007) creating a vertical gradient of temperature from bottom to top. Transference of heating promoted by acid vapors is responsible for the heating of the upper parts of vessels.

The possibility of losses by volatilization of compounds containing As, Cd, Pb, Se, and Zn in marine invertebrate samples was evaluated using cavity- and focused-microwave ovens employing acid digestion procedures. The choice of invertebrate marine samples for this study is based on the fact that many species are rich in As, Se and Pb compounds and these are prone to losses by volatilization. Invertebrate marine samples present large quantities of As, mainly as arsenobetaine, while Se is accumulated in soft tissues as seleniocytochrome. Concentrations of these elements may be used as indicators of contaminants in the surrounding water, based on the assumption that these metals accumulated in marine invertebrate tissues (Bellotto and Miekeley, 2007).

## 2. Materials and methods

### 2.1. Instrumentation

An inductively coupled plasma optical emission spectrometer with axially viewed configuration (ICP OES, VISTA PRO, Varian, Mulgrave, Australia) was used for As, Cd, Pb, Se, and Zn determination. The sample introduction system employed comprises a concentric nebulizer and a cyclonic spray chamber. The operational parameters employed are described in Table 1.

A focused-microwave oven with two cavities (Star System 2, CEM, Matthews, NC, USA) was employed for acid digestion of the samples. The heating program is shown in Table 2. A quartz reaction vessel was used in all digestions and the system reaction vessel plus condenser is 42 cm in height.

A cavity-microwave oven (Milestone, Sorisole, Italy) was used for closed vessel acid digestion of the samples. A 100 mL TFM<sup>®</sup> (modified polytetrafluoroethylene) closed vessel with calibrated resealing pressure relief mechanism was inserted on a rotating turntable inside the microwave oven cavity and the heating program was performed as described in Table 3.

### 2.2. Reagents, standards and samples

All solutions were prepared using deionized water. Hydrogen peroxide 30% v v<sup>-1</sup>, concentrated nitric and sulfuric acids (Merck, Darmstadt, Germany) were used for sample digestions. The multielement reference solutions of As, Cd, Pb, Se, and Zn for the analytical curve were prepared daily by suitable dilution from

**Table 1**

Instrumental parameters for elements determinations using ICP OES with axially viewed configuration.

Instrumental parameter	
Generator frequency (MHz)	40
RF power (kW)	1.3
Plasma gas flow (L min <sup>-1</sup> )	15.0
Auxiliary gas flow (L min <sup>-1</sup> )	1.5
Nebuliser gas flow-rate (mL min <sup>-1</sup> )	0.7
Sample flow-rate (mL min <sup>-1</sup> )	0.8
Analytical wavelengths (nm)	
As II	188.980
Cd II	214.439
Pb I	220.353
Se I	196.026
Zn II	213.857
Y II	371.029

**Table 2**

Heating program for focused-microwave-assisted acid-digestion of marine invertebrate samples. Initial addition: 2.5 mL concentrated H<sub>2</sub>SO<sub>4</sub> + 5.0 mL concentrated HNO<sub>3</sub>.

Step	t <sub>ramp</sub> (min)	T (°C)	t <sub>plateau</sub> (min)	Reagent (mL)	Aliquot (mL)
1	3.0	100	2.0	1.0 HNO <sub>3</sub>	1.0
2	3.0	150	2.0	1.0 HNO <sub>3</sub>	1.0
3	2.0	220	2.0	–	–
4	0	220	10.0	10.0 H <sub>2</sub> O <sub>2</sub>	1.0

**Table 3**

Heating program for cavity-microwave-assisted acid digestion of marine invertebrate samples.

Step	Power (W)	t <sub>ramp</sub> (min)	T (°C)	t <sub>plateau</sub> (min)
1	500	4.0	90	2.0
2	750	8.0	180	15.0
Ventilation	0	0	25	–

1000 mg L<sup>-1</sup> stock solutions (QHEMIS, High Purity, São Paulo, Brazil). All stock solutions were prepared in 5% v v<sup>-1</sup> HNO<sub>3</sub> medium. A stock solution containing 100 mg L<sup>-1</sup> of Y was used as internal standard in reference solutions and all digests obtained using focused-microwave oven before element determinations by ICP OES (Gouveia et al., 2001).

For this study, three species of marine invertebrate samples (i.e. mussel – *Mytella guyanensis*, oyster – *Crassostrea rhizophorae*, and clam – *Anomalocardia brasiliiana*) were collected in Todos os Santos Bay, Brazil. These marine invertebrates are very common and represent an important source of protein for Brazilian fishing communities. About 500 g of each species were collected manually, stored in plastic bags and refrigerated. In the laboratory, the invertebrate samples were cleaned with ultrapure water, the shells were open and the invertebrate tissues were separated and frozen. Afterwards, samples were freeze-dried and comminuted in a cryogenic mill. For the comminution a mass of 5 g of samples was inserted in a grinding vial, a polycarbonate cylinder supplied with two stainless steel plugs, immersed in liquid nitrogen and ground with an alternating magnetically driven steel impactor. Samples were precooled for 3 min and ground for 2 min. Three grinding cycles, with a cooling step of 1 min between cycles, were applied to each sample, totaling 18 min. The dried, homogenized samples were stored in polyethylene vessels. Care was taken during all steps of sample collection and pre-treatment to avoid contamination. The precision and accuracy were evaluated using a standard reference material from the National Institute of Standards Technology (NIST, Gaithersburg, MD, USA), oyster tissue SRM 1566b. To evaluate the agreement between obtained and certified

values a Student's *t*-test was applied. Glassware and plastic ware were soaked overnight in a 10% v v<sup>-1</sup> HNO<sub>3</sub>, and properly rinsed with deionized water before use.

### 2.3. Sample preparation

#### 2.3.1. Focused-microwave acid digestion procedure

A mass of about 250 mg of marine invertebrate samples was transferred to a microwave-vessel and the program described in Table 2 was performed with initial addition of 2.5 mL of sulfuric and 5.0 mL of nitric concentrated acids, without heating. In the first and second steps, after increasing the temperatures to 100 and 150 °C, respectively, an aliquot of 1.0 mL of HNO<sub>3</sub> 65% v v<sup>-1</sup> was automatically added in each step. In the last step, with a temperature of 220 °C, 10 mL of H<sub>2</sub>O<sub>2</sub> 30% v v<sup>-1</sup> was added in aliquots of 1 mL. Final volume was adjusted to 25 mL with deionized water up to the mark. The samples were prepared in triplicates, in independent analytical runs.

#### 2.3.2. Cavity-microwave acid digestion procedure

About 250 mg of marine invertebrate samples were transferred to TFM vessel. Then, 7 mL of HNO<sub>3</sub> 65% v v<sup>-1</sup> were added, and after 30 min contact, 1 mL of 30% v v<sup>-1</sup> H<sub>2</sub>O<sub>2</sub> was added. The vessels were closed and the heating program described in Table 3 was performed. The digests were transferred to 25 mL polypropylene vessels, which were filled up to the mark with deionized water. The samples were prepared in triplicates, in independent analytical runs.

## 3. Results and discussion

Results from focused-microwave oven were compared with those obtained after cavity-microwave oven (Table 4). The data obtained using the acid digestion in the cavity-microwave oven were assumed to represent a 100% recovery owing to the use of a cavity oven with closed vessel. The NIST SRM 1566b was used to check the accuracy and the results are shown in Table 5. It can be supposed that some As compounds present in marine invertebrate samples are not digested at relatively low temperatures (Karadjova

et al., 2007; Entwisle and Hearn, 2006; Matusiewicz and Stanisz, 2007); however, the chemical forms present in the digest probably did not exert a significant effect in ICP OES measurements. Results presented in Tables 4 and 5 indicated losses by volatilization after focused-microwave acid digestion. Table 4 shows that the recoveries for As, Cd, Pb, and Se varied from 64 to 83, 32 to 53, 12 to 44, and 65 to 96%, respectively. Zinc contents were higher in digestates from focused-microwave oven than cavity-microwave in oyster and clam samples. Lead and Cd were the elements that presented the highest percentual losses by volatilization in the studied samples. The high values obtained for Cd, Pb, and Zn can be explained by the influence of an inactive Pb smelter in the sediments of the sampling sites (Hatje et al., 2006).

The results indicated that losses by volatilization of some elements can be important for complex matrixes, such as marine invertebrates. As mentioned by Sturgeon and Mester (2002) losses of volatile elements are related to the complexity of the sample, e.g. the type of bonding between matrix compounds and elements.

In Table 5, it can be seen that results obtained with cavity-microwave-assisted acid digestion for As, Cd, and Zn were in agreement with the certified values at 95% confidence level, when applying Student's *t*-test. Lead and selenium were not detectable. Losses by volatilization were found for all investigated elements when using focused-microwave-assisted acid digestion compared to reference values.

Marine invertebrate samples were digested in a focused-microwave-oven using 2.5 mL H<sub>2</sub>SO<sub>4</sub>, 7.0 mL HNO<sub>3</sub>, and 10.0 mL H<sub>2</sub>O<sub>2</sub> and in the cavity-oven using only 7.0 mL HNO<sub>3</sub>, and 1.0 mL H<sub>2</sub>O<sub>2</sub>. Final adjusted volumes of the digests were the same. The final acidities in the focused-oven digests were about three times higher than in the cavity-oven due to the addition of sulfuric acid to the acid digestion mixture employed with the former system. In the focused-microwave-oven, the increase of temperature is critically dependent on the addition of H<sub>2</sub>SO<sub>4</sub> owing to the operation at about normal pressure. Consequently, addition of an internal standard to the digests is usually necessary to eliminate transport interferences when solutions are introduced by nebulization (Silva et al., 2002; Myers and Tracy, 1983; Korn et al., 2008).

It must be mentioned that considering the maximum temperature adopted in the focused-microwave acid digestion heating program, volatilization losses are expected for volatile elements and their compounds in marine invertebrate samples. However, tailored procedures could be employed for overcoming these problems. In a recent review (Nóbrega et al., 2006), some strategies were presented to use focused-microwave ovens associated to alkaline digestions and extractions. The focused-oven was applied for alkaline extraction of mono-, di-, and tributyltin in fish tissue using TMAH (Szpunar et al., 1996). An alkaline procedure using 20% of irradiation power (40 W) of a focused-oven for the extraction of Hg and butyltin compounds in samples of macrobenthos (Monperrus et al., 2005) was also developed. These procedures are easily implemented and allow the chemical speciation without any losses using digestions in alkaline medium at low irradiation power. However, this preliminary investigation about marine invertebrate samples suggests that for volatile elements and their respective compounds, such as As, Se and Pb, the best choice for sample acid digestion is the use of closed vessels in a cavity-microwave oven.

## 4. Conclusions

It was demonstrated that depending on the adopted digestion conditions, a focused-microwave oven operated at atmospheric pressure is prone to losses of volatile elements and compounds. The highest losses, up to 88%, were observed for Pb, and the lowest

**Table 4**  
Determination of As, Cd, Pb, Se, and Zn in marine samples after acid digestion in a closed- and in a focused-microwave oven (mean ± standard deviation; n = 9).

Samples	As (μg g <sup>-1</sup> )	Cd (μg g <sup>-1</sup> )	Pb (μg g <sup>-1</sup> )	Se (μg g <sup>-1</sup> )	Zn (μg g <sup>-1</sup> )
Oyster					
(1)	6.0 ± 0.9	2.9 ± 0.1	6.2 ± 0.1	6.2 ± 1.0	3294 ± 31
(2)	3.9 ± 0.2	1.6 ± 0.1	0.8 ± 0.1	4.2 ± 0.2	3607 ± 27
Mussel					
(1)	8.8 ± 0.7	2.5 ± 0.2	2.4 ± 0.2	7.3 ± 0.5	61 ± 1
(2)	7.1 ± 0.5	0.9 ± 0.1	1.1 ± 0.2	4.8 ± 0.6	60 ± 3
Clam					
(1)	8.8 ± 1.1	2.2 ± 0.2	5.1 ± 0.3	5.1 ± 0.3	55 ± 1
(2)	7.3 ± 0.8	0.7 ± 0.1	0.8 ± 0.1	4.9 ± 1.3	61 ± 1

(1) Cavity-microwave oven; (2) focused-microwave oven.

**Table 5**  
Determination of As, Cd, Pb, Se, and Zn in NIST SRM 1566-Oyster tissue after acid digestion in a cavity- and in a focused-microwave oven (mean ± standard deviation; n = 9).

	As (μg g <sup>-1</sup> )	Cd (μg g <sup>-1</sup> )	Pb (μg g <sup>-1</sup> )	Se (μg g <sup>-1</sup> )	Zn (μg g <sup>-1</sup> )
Certified values	7.7 ± 0.7	2.5 ± 0.1	0.31 ± 0.01	2.1 ± 0.2	1424 ± 46
Cavity-oven	7.3 ± 1.1	2.4 ± 0.1	<0.009	<0.010	1369 ± 24
Focused-oven	<0.009	1.1 ± 0.02	<0.009	<0.010	1213 ± 19

< = <Limit of detection (LOD).

losses were observed for Zn. The main recommendation derived from this study is the need to carefully consider the use of focused-microwave-assisted digestion taking into account the behavior of sample constituents, especially volatile elements. Losses and contaminations are critical aspects that must be fully evaluated when working with environmental samples.

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