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Focused Microwave-Induced Combustion: A New Technique for Sample Digestion

Márcia F. Mesko,[†] Juliana S. F. Pereira,[‡] Diogo P. Moraes,[‡] Juliano S. Barin,[‡] Paola A. Mello,[‡] José Neri G. Paniz,[‡] Joaquim A. Nóbrega,[§] Maria G. A. Korn,[||] and Erico M. M. Flores*[,†]

Instituto de Química e Geociências, Universidade Federal de Pelotas, 96010-610, Pelotas, RS, Brazil, Departamento de Química, Universidade Federal de Santa Maria, 97105-900, Santa Maria, RS, Brazil, Departamento de Química, Universidade Federal de São Carlos, 13565-905, São Carlos, SP, Brazil, and Departamento de Química Analítica, Instituto de Química, Universidade Federal da Bahia, 40170-290, Salvador, BA, Brazil

A procedure for sample digestion based on focused microwave-induced combustion (FMIC) is proposed. This system was developed using a commercial focused microwave oven with a lab-made quartz sample holder and a modified glass vessel. Oxygen flow was used to start and support the combustion. A botanical sample was used to evaluate the operational conditions for further Al, Ba, Ca, Fe, Mg, Mn, Sr, and Zn determination by inductively coupled plasma optical emission spectrometry. Pelletized samples were positioned on the quartz holder, and 50 μL of 6 mol L^{-1} NH_4NO_3 solution was added as igniter. Combustion was completed in less than 2 min, and the temperature was higher than 950 °C. The use of a reflux step, the position of sample holder inside the vessel, sample mass, ignition and combustion time, oxygen flow rate, and condenser type were evaluated. Results were compared with those obtained by focused microwave-assisted wet digestion and by high pressure microwave-assisted wet digestion. Agreement of 95−103% was obtained for certified reference materials digested by FMIC (reflux step with 10 mL of 4 mol L^{-1} HNO_3). With the proposed procedure, a complete sample decomposition (residual carbon content lower than 0.5%) was achieved with low consumption of reagents as only 10 mL of diluted nitric acid was necessary. Low relative standard deviation (lower than 3.8%) was observed and high amount of sample (up to 1500 mg) could be digested that allowed lower limits of detection.

Microwave-assisted procedures are well-established strategies currently used in routine analysis for different sample preparation steps, such as drying, extraction, and digestion.[1,2] In general, microwave-assisted digestion can be carried out using cavity or focused microwave ovens. In cavity-microwave ovens, closed vessels are currently used, allowing digestions at high pressure and temperature resulting in low residual carbon contents (RCC).[3−5]

In spite of the good performance of cavity-microwave ovens, the use of focused microwave radiation could be advantageous in situations that require the digestion of higher amounts of organic sample, resulting in the generation of a huge amount of gas, or when multiple additions of concentrated acids are required during digestion.[6,7] Additionally, other features may be pointed out as better safety conditions due to the operation at atmospheric pressure, possibility of using several types of materials for the reaction vessels, such as borosilicate glass, quartz, and PTFE, programmable addition of reagents or samples during digestion, and simultaneous control of different heating programs for different samples owing to the possibility of operating each reaction vessel independently.[6,8]

One of the main drawbacks of focused microwave systems is related to the high amounts of concentrated acids, especially sulfuric acid, that is necessary to reach high temperatures at atmospheric pressure, which may increase blank values. Moreover, the presence of high acid concentrations in digests may not be supported by some analytical techniques, such as inductively coupled plasma optical emission spectrometry (ICP OES), inductively coupled plasma mass spectrometry, ion chromatography, or electroanalytical techniques, and a subsequent step to either remove or dilute the residual acid is generally necessary.[9−11] This aspect can be overcome by changing the usual procedure that recommends the programmed addition of concentrated acids to

* Corresponding author. Fax: +55 55 3220-9445. E-mail address: flores@quimica.ufsm.br.

[†] Universidade Federal de Pelotas.

[‡] Universidade Federal de Santa Maria.

[§] Universidade Federal de São Carlos.

[||] Universidade Federal da Bahia.

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the sample or by addition of sample aliquots gradually to the acid medium. It was proved that this alternative procedure is effective for digesting large amounts of samples using lower volumes of concentrated acids.[12,13]

Combustion techniques had a spread use in the past, mainly due to the high efficiency of sample oxidation, digestion of relatively high sample masses and the suitable purity of oxygen when it is compared to the concentrated acids used in conventional wet digestion procedures. Different combustion systems in open vessels have been proposed for trace element analysis, e.g. low temperature ashing, Wickbold combustion, and the Trace-O-Mat system.[14−16] However, the most successful combustion techniques were those performed in closed vessels as oxygen flask combustion and combustion bombs.[14−17] In these techniques, sample is burnt in the presence of an oxygen excess and the combustion products are absorbed in a suitable solution into the same reaction vessel.[15,16] In addition, a fast and complete combustion/digestion is performed in few seconds with minimum reagent consumption.

Recently, microwave-induced combustion (MIC) was proposed in order to combine the advantages of classical combustion techniques in closed vessels with those from conventional closed systems assisted by microwave radiation.[16] For this technique, organic samples are combusted in closed quartz vessels pressurized with oxygen and an ignition step is performed under microwave radiation using cavity-ovens. A small quartz holder, placed inside the quartz vessel, is the only necessary change in relation to the conventional apparatus originally designed for conventional microwave-assisted digestion in closed vessels. In addition, this system allows one to perform a subsequent reflux step when needed that is not available in other combustion techniques as oxygen flask and combustion bombs.[18] Microwave-induced combustion has been applied for digestion of biological samples,[18−20] carbon black-containing elastomers,[21] coal,[22] and for crude oil and crude oil products digestion[23−27] for both metal and nonmetal determinations.

In spite of the good efficiency of MIC to digest samples with complex matrix and its suitability for further metals determination using diluted acids, the amount of sample that can be burnt is limited to 500 mg due to the high pressure generated inside the closed vessel.[16] In this sense, if a sample preparation system could allow the combination of the advantages of MIC technique with those of focused microwave assisted digestion, it should be advantageous for the sample preparation field due to the possibility to digest higher amount of samples in addition to a lower acid consumption and better digestion efficiency.

In this work, focused microwave-induced combustion (FMIC) is proposed for the first time as a sample preparation technique to overcome the limitations of MIC concerning the digestion of high sample masses (up to 1500 mg) and to improve the digestion efficiency in comparison with focused microwave-assisted wet digestion in closed vessels, especially concerning to the high amounts of acids generally required. The effect of a reflux step, the sample quartz holder position inside the vessel, ignition time, combustion time, oxygen flow rate, and sample mass were studied. The type of condenser and glass vessel were also investigated. In order to demonstrate the potential for sample preparation, the proposed FMIC system was applied to the digestion of botanical samples and results were compared with those obtained using focused microwave-assisted wet digestion (FMAWD) and high-pressure microwave-assisted wet digestion (MAWD) in closed vessels. The determination of Al, Ba, Ca, Fe, Mg, Mn, Sr, and Zn was chosen as application examples and determinations were performed by ICP OES. Accuracy was evaluated using certified reference materials (CRM) of plants.

EXPERIMENTAL SECTION

Instrumentation. A commercial microwave oven with focused microwave radiation with two cavities (Star System 2, 800 W, CEM, Matthews, NC) equipped with glass vessels of 180 mL of capacity and lab-made condensers was used for FMIC studies. The focused microwave works at atmospheric pressure and allows independent temperature control in each cavity. For ignition step in the FMIC procedure, the maximum microwave power was applied (800 W) and the maximum temperature was set at 430 °C. The focused microwave oven was equipped with a vapor collection and automatic reagent addition modules (CEM, model reagent addition EX4, model number 560980). This equipment was also used for procedures by FMAWD.

In order to allow sample combustion using the proposed FMIC system, the original glass vessels were modified to allow an oxygen flow entrance and the introduction of a sample quartz holder inside the vessel (Figure 1). A water-cooled condenser was used instead of the conventional air-cooled system provided by the manufacturer. The new design of condenser has a higher surface for better cooling of vapors. Cooling was performed using water (15 °C). The height of sample quartz holder inside the vessel was optimized to maintain it in a position of maximum microwave radiation incidence. The sample quartz holder used for sample combustion was designed in order to reduce the cool surfaces

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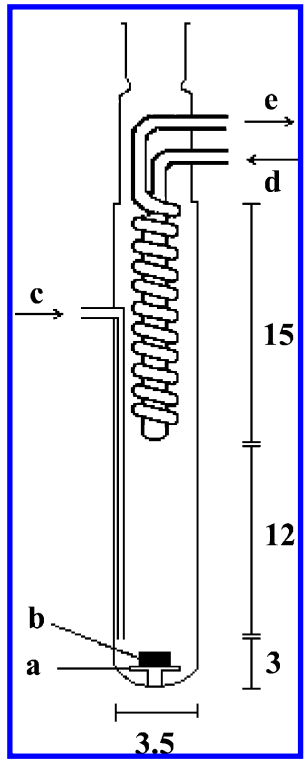

Figure 1. Focused microwave-induced combustion flask (dimensions in centimeters): (a) quartz sample holder, (b) pelletized sample, (c) entrance of oxygen, (d) condenser water inlet, (e) condenser water outlet.

that might cause carbon deposits during combustion. It was performed by making the bottom part of quartz holder with four small slits.

For comparison of results, samples were digested by conventional FMAWD (CEM system) and also in a pressurized closed system by MAWD using a microwave sample preparation system (Multiwave 3000, Anton Paar, Graz, Austria) equipped with eight high-pressure quartz vessels. For MAWD, vessels with 80 mL of internal volume and maximum pressure and temperature of 8 MPa and 280 °C, respectively, were used.

Analytes and residual carbon content (RCC) were determined using a simultaneous inductively coupled plasma optical emission spectrometer (Spectro CIROS CCD, Spectro Analytical Instruments, Kleve, Germany) with axial view configuration. Argon with purity of 99.996% (White Martins−Praxair, São Paulo, SP, Brazil) was used for plasma generation, nebulization and as auxiliary gas. The selected wavelengths used for Al, Ba, Ca, Fe, Mg, Mn, Sr, and Zn determinations and operational parameters are listed in Table 1, and they were used as recommended by the instrument manufacturer.[28] Residual carbon content was also determined in digests by ICP OES after FMIC, FMAWD, and MAWD at 193.091 nm using the conditions described in previous work.[22]

The temperature during sample combustion was determined using an optical pyrometer (Ultimax Infrared Thermometer, model Ultimax 20, Ircon, Niles, IL) equipped with a close-up VX-CL1 lens.

Samples, Reagents, and Standards. Commercial medicinal plant (*Peumus boldus sp.*, SiO_2 content below 1%), was used to optimize the operational conditions for FMIC. Samples were

(28) *Spectro Ciros CCD*; software version 01/March 2003, Spectro Analytical Instruments GmbH & Co. KG: Kleve, Germany, 2003.

Table 1. Operational Conditions for ICP OES[a]

parameter	ICP OES
spray chamber	Scott-type
nebulizer	cross flow
view	axial
RF power (W)	1400
Ar plasma flow rate (L min^{-1})	12
Ar auxiliary flow rate (L min^{-1})	1.0
Ar nebulizer flow rate (L min^{-1})	1.0
analytical wavelenghts (nm)	
Al II	167.078
Ba II	455.404
Ca II	396.847
Fe II	259.940
Mg II	279.553
Mn II	257.611
Sr II	407.771
Zn II	213.857
C I	193.091

[a] RCC measurements were performed according to ref 22.

heated at 60 °C in an oven (model 400/2ND, Nova Etica, São Paulo, Brazil) and ground using a cryogenic mill (Spex Certiprep, Model 6750, Metuchen, NJ) using a precooling time of 5 min followed by 2 min for grinding. This procedure was repeated using 1 min of recooling between grinding steps. Certified reference materials provided by National Institute of Standards and Technology (NIST, Gaithersburg, MD), apple leaves (NIST 1515), and peach leaves (NIST 1547) were used for accuracy evaluation of the proposed procedure. Samples and CRM's were pressed as pellets (diameter of 13 mm) using a hydraulic press set at 3 ton by 1 min (Specac, Orpington, UK) for digestion by FMIC.

Analytical-grade reagents provided by Merck (Merck, Darmstadt, Germany) were used to prepare all solutions and standards. Water was purified using a Milli-Q system (18.2 MΩ cm, Millipore, Bedford, MA). Concentrated nitric acid was distilled in a subboiling apparatus (model DuoPur, Milestone, Sorisole, Italy). Reference standard solutions were prepared before use by dilution of multielement stock reference solutions containing 10 mg L^{-1} analytes (PlasmaCal calibration solution SCP33MS, SCP Science, Quebec, Canada) in 5% (v/v) HNO_3. A stock reference solution for RCC determination was prepared by dissolution of citric acid in water. Concentrated nitric acid was used for FMAWD and MAWD. Diluted nitric acid was used as absorbing solution in FMIC procedure. Sulfuric acid and hydrogen peroxide (30% (w/w), Synth, Diadema, Brazil) were used for FMAWD.

A small disk of filter paper (15 mm of diameter, 12 mg) with low ash content (Black Ribbon Ashless, Schleicher & Schuell GmbH, Dassel, Germany) and an ammonium nitrate solution (6 mol L^{-1}, salt from Merck) were used as igniter for the combustion process by FMIC. Filter paper was previously cleaned with 10% (v/v) HNO_3 for 20 min in an ultrasonic bath, rinsed with water, and dried in a Class 100 laminar flow bench (CFLH-12, Veco, Brazil) before use. Glass and quartz materials were soaked in 10% (v/v) HNO_3 for 48 h and thoroughly washed with water before use. Quartz vessels used in MAWD were cleaned with 6 mL of concentrated HNO_3, for 10 min at 1400 W. Vessels used in FMAWD and FMIC (with quartz

holders) were cleaned with 15 mL of concentrated HNO_3, for 10 min at 130 °C.

Digestion of Botanical Samples Using Focused Microwave-Assisted Wet Digestion and High-Pressure Microwave-Assisted Wet Digestion. Samples of plants (*Peumus boldus*) were previously digested using FMAWD. Decomposition was performed as recommended in CEM Application Number SS08 (ID-2) for citrus sample, which uses the commercial air-cooled condenser, 1 g of sample, 2.5 mL of concentrated sulfuric acid, 12 mL of concentrated nitric acid, and 20 mL of hydrogen peroxide (30% w/w).[29] This procedure was changed to digest 1.5 g of sample, using the same water-cooled condenser used for FMIC and reagents were proportionally increased to this sample mass. The heating program was as follow: (i) ramp of 3 min up to 130 °C; (ii) 1 min at 200 °C (ramp of 1 min); (iii) 5 min at 250 °C (ramp of 1 min); and (iv) 10 min at 200 °C (H_2O_2 is added only before this step). After digestion, samples were diluted with water to 50 mL in polypropylene vessels for analysis by ICP OES.

Samples were also digested in closed vessels by MAWD. Microwave heating program was performed as described by instrument manufacturer for plant digestion, using 6 mL of concentrated HNO_3 and 500 mg of sample.[30] After digestion, the resulting solution was transferred to 30 mL polypropylene vials and diluted with water. Analytes in final digests were measured by ICP OES.

Proposed Focused Microwave-Induced Combustion Procedure. Operational conditions were optimized using sample pellets ranging from 100 to 1500 mg. Pellets were placed together with the filter paper on the quartz holder device specially designed for the FMIC procedure. Then, 50 μL of 6 mol L^{-1} NH_4NO_3 solution was added to the filter paper,[22] and the quartz holder was introduced inside the quartz vessel. The vessel was placed inside the focused microwave oven, and the condenser was connected to the upper part. A continuous flow rate of oxygen (2 L min^{-1}) was passed inside the vessels by a side entrance (Figure 1). The microwave heating program was immediately started: (i) 5 s at maximum power for sample ignition and (ii) microwave radiation was interrupted (after sample ignition) and oxygen flow rate was increased from 2 to 15 L min^{-1}. After 10 min (cooling step), 10 mL of absorbing solution (4 mol L^{-1} HNO_3) were automatically added inside the glass vessel using the reagent addition module, and a reflux step was performed under focused microwave radiation, at 125 °C for 5 min. Digests were diluted to 30 mL with water.

RESULTS AND DISCUSSION

Development of Focused Microwave-Induced Combustion System. Initial studies of FMIC were performed in order to evaluate the height of the sample quartz holder inside the glass vessel. The adjustment of the height is essential to insert the sample pellet at the position of maximum microwave radiation incidence. Therefore, preliminary studies were performed using the quartz holder device positioned in different heights: 7, 10, and 13 mm off the bottom part of glass vessels. Pellets with masses of about 200 mg and reflux step for 5 min were arbitrarily used in this step. The sample pellet and filter paper were positioned in the quartz holder device and 50 μL of a 6 mol L^{-1} NH_4NO_3 solution was added to the paper. Microwave irradiation was started, and oxygen was kept flowing inside the vessel. After ignition of the sample pellet (about 5 s), microwave irradiation was stopped. When the absorbing solution was added inside the vessel prior to the combustion step and the microwave radiation was started, the solution evaporated before the ignition and the paper and sample became too wet avoiding the further combustion. In this sense, the ignition step was carried out without any solution inside the vessel and the absorbing solution was added after finishing the combustion process.

It was observed that combustion was not reproducible, i.e. around 70% of failure for 10 experiments when the quartz holder was positioned about 13 mm of the bottom part of the glass vessel. On the other hand, when the position of sample quartz holder was changed to 10 mm, combustion occurred in 50% of the experiments (n = 10). Positioning the quartz holder at 7 mm presented better efficiency of sample combustion and combustion always occurred, since it was the position of maximum microwave radiation incidence.

Further studies were performed in order to evaluate the maximum sample mass that could be combusted by FMIC. Sample pellets from 100 to 1500 mg were positioned inside the sample quartz holder and combustion was carried out using 2 L min^{-1} of oxygen (this flow rate was arbitrarily set for preliminary experiments). It was verified that the combustion of sample masses higher than 200 mg was not complete and some solid residues and smoke were observed probably due to the low oxygen flow rate. The oxygen flow rate necessary for starting and keeping the ignition step was evaluated from 2 to 15 L min^{-1}. Using an initial oxygen flow rate higher than 5 L min^{-1}, it was verified that combustion did not occur probably because the filter paper became dry and the combustion process did not start. Taking into account that the ignition process always occurred when 2 L min^{-1} of O_2 was used this condition was set for further tests.

For complete combustion of masses higher than 200 mg, the oxygen flow rate was increased to 15 L min^{-1} immediately after the ignition. In this condition, the combustion proceeds as a self-sustaining process even without the incidence of microwave radiation. Then, combustion time for a pellet containing 1500 mg of botanical sample was between 80 and 120 s. Measurements using an infrared thermometer showed that temperature achieved during the combustion was always higher than 950 °C for 1500 mg of botanical sample. The emission of a white and bright radiation during the whole combustion process was also an indicative about the high temperature achieved. However, despite the high temperature, no damages were observed in the holders and vessels.

After combustion of samples by FMIC, the reflux step was performed by adding 10 mL of 4 mol L^{-1} HNO_3 using the reagent addition module and the commercial air cooled condenser to evaluate the analyte recoveries. It was observed that the absorbing solution was quickly evaporated and after 1 min, the

(29) CEM Application Note Citrus Leaves SS08 (ID2). http://www.cem.com/download52.html (accessed Dec 2009).

(30) *Microwave Sample Preparation System*; software version v1.27-Synt, Anton Paar GmbH: Graz, Austria, 2003.

Table 2. Determination of Metals by ICP OES in a *Peumus boldus sp.* Sample Using FMIC ($n = 3$, Mean ± Standard Deviation, 10 mL of 4 mol L^{-1} HNO_3 as Absorbing Solution, Reflux Step, 1500 mg of Sample) and Comparison with Microwave-Assisted Digestion in a High Pressure System (MAWD) and in an Open System (FMAWD)

	concentration, $\mu g\ g^{-1}$			limit of detection, $\mu g\ g^{-1}$		
element	MAWD	FMAWD	FMIC	MAWD	FMAWD	FMIC
Al	2.41 ± 0.24	2.41 ± 0.32	2.40 ± 0.09	0.30	0.40	0.09
Ba	<0.3	<0.1	<0.05	0.30	0.10	0.05
Ca	670 ± 28	710 ± 35	673 ± 23	0.06	0.10	0.03
Fe	4.38 ± 0.19	4.43 ± 0.28	4.69 ± 0.15	0.02	0.05	0.01
Mg	839 ± 30	908 ± 55	875 ± 28	0.04	0.03	0.02
Mn	13.6 ± 0.7	14.6 ± 0.8	13.3 ± 0.4	0.03	0.02	0.01
Sr	2.27 ± 0.12	2.53 ± 0.10	2.33 ± 0.07	0.06	0.03	0.01
Zn	2.77 ± 0.17	2.80 ± 0.25	2.76 ± 0.10	0.09	0.06	0.03

vessel was completely dry. Therefore, the use of the commercial air-cooled condenser was not suitable for the FMIC procedure and the reflux step was not suitable with this type of condenser. Thus, a water-cooled condenser was specially designed (Figure 1), and it was verified that the absorbing solution was completely retained inside the vessel even after 5 min of reflux. This lab-made condenser was used for further studies.

Absorbing Solution and Reflux Step of FMIC. The FMIC procedure is performed in an oxygen rich atmosphere and the main combustion products are oxides of analytes. Consequently, the absorbing solution must be able to bring these products into solution before determination using ICP OES. It was already demonstrated that the absorbing solution has an important role because the type and the concentration of the solution are not the same for different analytes as well as the time for absorption.[16]

In this work, reflux step was performed using 5 min and 4 mol L^{-1} HNO_3. This solution was chosen taking into account the previous works using MIC for similar analytes.[19,21,23] A volume of 10 mL of absorbing solution was used in order to put the bottom part of sample holder into the solution. The use of a reflux step can ensure a better washing of sample holder and internal parts of vessel and condenser and quantitative recoveries were achieved for all analytes. Final digests were diluted to 30 mL with water leading to a ca. 1.3 mol L^{-1} nitric acid concentration that is suitable for sample introduction by ICP OES using pneumatic nebulization. A single washing of vessel and sample holder with 10 mL of 4 mol L^{-1} HNO_3 after combustion, without 5 min of microwave irradiation, resulted in nonquantitative recoveries and relative standard deviation (RSD) between 25 and 40%. Therefore, a reflux step with 4 mol L^{-1} HNO_3 was used for further studies using FMIC.

The residual carbon content was determined to evaluate the efficiency of combustion process. Using FMIC, the RCC values were below 0.5% for samples and CRMs. The low RCC obtained with the proposed procedure can be related to the high temperature reached during the combustion in an oxygen rich atmosphere. The RCC in digests obtained by FMAWD were between 7 and 14%, even using sulfuric acid and high temperatures during the procedure. Therefore, the proposed system was efficient to destroy practically all the organic matrix avoiding the use of concentrated acids.

Determination of Metals in Botanic and CRM Samples after FMIC Procedure. *Peumus boldus sp.* sample used in this work was analyzed using the chosen conditions for FMIC and further determination by ICP OES using 1500 mg of sample. This sample was previously characterized by two independent digestion procedures (FMAWD and MAWD) using ICP OES determination (Table 2). No statistical difference (*t*-test, confidence level of 95%) was observed for results using both digestion procedures. The proposed FMIC gave results in good agreement with those using FMAWD and MAWD. It is important to point out that the standard deviation (SD) for FMIC was lower than the other digestion procedures. It can be related to the higher sample mass that could be used in FMIC system (1500 mg in comparison with 500 mg for MAWD). As the same sample mass could be used in FMAWD (1500 mg), a similar SD could be expected. However, the higher SD observed for FMAWD in comparison to FMIC can be associated to the higher volume of concentrated reagents (more than 30 mL per sample) necessary for digestion. For FMIC, as only 10 mL of diluted HNO_3 was used to ensure quantitative recoveries the SD was even better than the other procedures.

As the proposed FMIC procedure allows the use of a relatively high sample mass, the limits of detection (LOD) would be expected to be improved in comparison to the other procedures. In addition, for FMIC the blank values were lower than those obtained by FMAWD and LODs obtained for FMIC (Table 2) were improved up to a factor of 2−3. It is an important aspect for some analytes typically present in low concentrations in samples and always prone to contamination problems (especially for Zn). In addition, sulfuric acid and hydrogen peroxide were used for FMAWD and they could increase the contamination by the reagents, resulting in higher blank values. Table 3 shows some characteristics related to proposed FMIC system, and a comparison to FMAWD and MAWD. It is possible to see some advantages of the proposed procedure as lower RCC and lower reagents consumption.

Table 3. Comparison of the Main Characteristics of the Proposed FMIC, FMAWD, and MAWD

parameter	FMIC	FMAWD	MAWD
maximum sample mass (mg)	1500	1500	500
reagents	4 mol L^{-1} HNO_3 (10 mL)	14 mol L^{-1} HNO_3 (17 mL) 18 mol L^{-1} H_2SO_4 (3 mL) 30% (w/w) H_2O_2 (30 mL)	14 mol L^{-1} HNO_3 (6 mL)
final digestion volume (ml)	30	50	30
RCC (%)	<0.5	7−14	4−6
digestion time (min)	20	25	40
RSD (%)	3.0−3.8	3.9−13.3	3.6−10.0

Table 4. Determination of Metals by ICP OES in Certified Reference Materials Using FMIC Procedure (Mean ± Standard Deviation, $n = 3$, 10 mL of 4 mol L^{-1} HNO_3 Absorbing Solution, Reflux Step, Sample Mass of 500 mg)

	certified value, $\mu g\ g^{-1}$		concentration, $\mu g\ g^{-1}$	
element	apple leaves	peach leaves	apple leaves	peach leaves
Al	286 ± 9	249 ± 8	291 ± 10	248 ± 9
Ba	49 ± 2	124 ± 4	48.3 ± 1.8	126 ± 4
Ca	1.526 ± 0.015	1.56 ± 0.02	1.529 ± 0.052	1.55 ± 0.04
Fe	83 ± 5	218 ± 14	85.4 ± 2.9	207 ± 6
Mg	0.271 ± 0.008	0.432 ± 0.008	0.276 ± 0.010	0.436 ± 0.016
Mn	54 ± 3	98 ± 3	52.8 ± 1.9	96.8 ± 3.2
Sr	25 ± 2	53 ± 4	24.3 ± 0.9	54.6 ± 2.0
Zn	12.5 ± 0.3	17.9 ± 0.4	12.8 ± 0.4	17.2 ± 0.5

Accuracy was evaluated by using CRM of apple leaves and peach leaves. Table 4 shows the results for apple and peach leaves samples applying the established conditions for FMIC and ICP OES measurements. All results were in agreement of 95 to 103% for CRMs. Despite the use of a partially open vessel (typical of focused microwave ovens), blanks for FMIC were always low, probably due to the better cleaning of holders and vessels during reflux step and also by the use of a diluted nitric acid solution as absorbing medium.

CONCLUSIONS

This study demonstrated the feasibility of performing microwave-induced combustion using focused microwave radiation. The main advantages of FMIC are the possibility to digest relatively higher sample masses, up to 1500 mg, using diluted nitric acid solution as absorbing medium. In addition, as a result of higher sample mass the RSD was lower than those obtained for other procedures. The proposed procedure was suitable for determining hardly extracted elements in botanical samples, such as aluminum, iron, and calcium. The purpose of the present work was to demonstrate the basic concept of FMIC procedure and experiments with further applications are in progress.

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