A simple design for microwave assisted digestion vessel with low reagent consumption suitable for food and environmental samples

Mehrdad Gholami1,2, Shima Behkami2, Sharifuddin Md. Zain2 & Sezgin Bakirdere3

The objective of this work is to prepare a cost-effective, low reagent consumption and high performance polytetrafluoroethylene (PTFE) vessel that is capable to work in domestic microwave for digesting food and environmental samples. The designed vessel has a relatively thicker wall compared to that of commercial vessels. In this design, eight vessels are placed in an acrylonitrile butadiene styrene (ABS) holder to keep them safe and stable. This vessel needs only 2.0 mL of HNO3 and 1.0 mL H2O2 to digest 100 mg of biological sample. The performance of this design is then evaluated with an ICP-MS instrument in the analysis of the several NIST standard reference material of milk 1849a, rice flour 1568b, spinach leave 1570a and Peach Leaves 1547 in a domestic microwave oven with inverter technology. Outstanding agreement to (SRM) values are observed by using the suggested power to time microwave program, which simulates the reflux action occurring in this closed vessel. Taking into account the high cost of commercial microwave vessels and the volume of chemicals needed for various experiments (8–10 mL), this simple vessel is cost effective and suitable for digesting food and environmental samples.

Most trace analytical techniques need a homogeneous liquid sample to operate. Therefore, solid samples should be completely dissolved and digested prior to analysis. On the other hand, the digestion step is the Achilles’ heel of trace element analysis. If the digestion technique is too gentle, it will not break the sample matrix, therefore, the target analytes will not be released into the solution1. However, a method that is too harsh will not only destroy the sample matrix, but might also cause the loss of the analyte of interest. This is particularly the case with metals such as antimony, arsenic or tin1.

One of the techniques that is often used for digestion is ashing, which requires burning of the sample matrix until it becomes ash. The ash is usually soluble in an acid solution. This technique may also cause loss of analytes at high temperatures and yield poor results. Another difficulty is that some matrices do not easily turn into ash and may not even completely burn. Therefore, some of the target elements may not be taken into the solution1–3.

Fusion is another technique used for difficult matrices. This technique is very labor intensive and could be very expensive; furthermore, the high salt load and contamination problems due to the fluxing agent are the main disadvantages of this technique1–3.

Microwave digestion technique is an efficient, fast, and reproducible sample preparation method. In this method, the reaction timescale is dramatically reduced. Moreover, the potential for contamination decreases compared to open digestion techniques1–12. The first application of microwave in sample preparation was reported in 1975. In this study, a domestic microwave oven was employed to digest biological samples in an Erlenmeyer flask13. Soon after, scientists applied closed vessels to digest samples in higher temperatures and pressures14–24. All the closed vessels are armed with a safety system, which is either a safety membrane or a safety valve that opens instantly to control any increase in pressure by releasing some of the gases1. Therefore, volatile elements may be lost when gases are released from the closed vessels and this could be a reason for the lower recoveries in digestion1.

1Department of Chemistry, Marvdasht Branch, Islamic Azad University, P.O. Box 465, Marvdasht, Iran. 2Department of Chemistry, University of Malaya, Kuala Lumpur 50603, Malaysia. 3Department of Chemistry, Yildiz Technical University, 34220 Istanbul, Turkey. Correspondence and requests for materials should be addressed to M.G. (email: mehrdad897@yahoo.com or gholami@miau.ac.ir)
The amount of chemical reagent typically used for digestion of biological samples with commercial vessel/microwave in other studies is about 8–10 mL; nevertheless, in this study, a maximum of 3.0 mL of chemical reagents are needed (2.0 mL of nitric acid and 1.0 mL of hydrogen peroxide).

Furthermore, the aim of this research was to design a simple PTFE vessel, which is safe to be used in a domestic microwave, as commercial ones might not be available for all due to its high cost. Finally, the performance of this design is evaluated with an ICP-MS instrument by analyzing the milk powder NIST 1849a, rice flour NIST 1568b, Peach Leaves NIST 1547 and Spinach Leaves NIST 1570a.

Results
To evaluate the accuracy and applicability of the designed vessel in the proposed power to time program, the SRMs of milk powder, rice flour, peach and spinach leaves were digested and analyzed. The results found for these SRMs are compared with their corresponding certified values and reported in Tables (1–4).

Discussion
The results observed in Tables 1 and 2 shows excellent recoveries for most of the elements that exist in the milk powder and rice flour SRM samples. Nevertheless, the results shown in Tables 3 and 4 for the analysis of peach and spinach leaves SRMS, respectively revealed that the recoveries for Al, Cu, Zn, V, and Ni are not as good as the other elements. The lower recovery in this case might be contributed to the silicon content of peach and spinach leaves; these elements are not completely released in microwave digestion as jelly silicon contents can keep part of the analytes while digestion is in progress. This reduction in the recovery of elements, which was mentioned above, is directly related to the amount of silicon content of the leaves. The mean silicon content of peach NIST 1547 and spinach NIST 1570a leaves has been reported to be 979 and 1137 mg kg$^{-1}$, respectively. It is clear that lower recoveries for Al, Cu, Zn, V, Mo, and Ni observed in case of the peach and spinach leaves are caused by their higher content of silicon.

As explained, in order to prevent over pressure condition in the designed vessel, a domestic microwave oven was used, facilitated with inverter technology. In conventional microwave ovens, the power of microwave is
always the maximum nominal power of the oven. Hence, for generating lower powers of radiation during the
time of operation, the device was turned on and off successively so that the approximate required power of radi-
ation can be transferred into the object. Nevertheless, in the microwave oven with inverter technology the exact
required power of radiation can be applied continuously on the target.

The amount of chemical reagent typically used for digestion of biological samples is about 8–10 mL25,26; how-
ever, in this study, a maximum of 3.0 mL of chemical reagents (2.0 mL of nitric acid and 1.0 mL of hydrogen per-
oxide) were used to digest 0.10 g of milk powder, rice flour, spinach and peach leaves SRMs. The recommended
procedure for the digestion of SRM 1849 – Infant/Adult Nutritional Formula is digestion of 1.00 g of this sample
with 10.0 mL of HNO3 for the 75 mL MARSXpress vessel28. The results reported by the CEM corporation for this
SRM are given in Table 5.

As it is clear the Agreement to (SRM) values (%) reported by recommended procedure of CEM corporation
are relatively poorer than the results obtained in this study. This can be attributed to the amount of (SRM) sam-
ple, which is 1.0 g, and the amount of recommended HNO3, which is 10.0 mL. This amount of HNO3 and (SRM) sam-
ple can cause overpressure in the digestion vessel and activation of safety valve. Therefore, because of releasing
over-pressurized gases, some of the target samples will be lost and this is the reason for poorer results reported
by CEM corporation.

Conclusions
A simple microwave digestion vessel design was introduced for digesting some food and environmental samples;
the accuracy and applicability of this design was evaluated by the analysis of milk powder, rice flour, peach leaves,
and spinach leaves SRMs. In this study a power to time microwave program, which simulates the reflux action
occurring in this closed vessel were applied. Acceptable agreement to certified values have been observed for
the analysis of milk powder and rice flour SRMs for most elements. However, it was observed that agreement to
(SRM) values for some elements in peach and spinach leaves SRMs were not as good as milk powder and rice
flour SRMs. It is concluded that this reduction in the recoveries as observed in some of the elements could be
attributed to the silicon contents of these leaves. Moreover, the amount of chemicals used for the digestion in this

| Elements | Concentration (mg.kg⁻¹) | RSD% | Agreement to (SRM) values (%) |
|----------|--------------------------|------|-------------------------------|
| Al       | 249 ± 8                  | 1.56 | 94.8                          |
| As       | 0.060 ± 0.018            | 11.6 | 100                           |
| Cd       | 0.026 ± 0.003            | 3.7  | 103.8                         |
| Cu       | 3.7 ± 0.4                | 1.47 | 97.3                          |
| Fe       | 218 ± 14                 | 2.1  | 98.2                          |
| Mn       | 98 ± 3                   | 1.25 | 98.1                          |
| Mo       | 0.060 ± 0.008            | 3.44 | 96.7                          |
| Ni       | 0.69 ± 0.09              | 11.1 | 104.3                         |
| Pb       | 0.87 ± 0.03              | 3.48 | 98.8                          |
| Se       | 0.120 ± 0.009            | 6.66 | 100                           |
| Sr       | 53 ± 4                   | 3.7  | 101.8                         |
| V        | 0.37 ± 0.03              | 5.71 | 94.6                          |
| Zn       | 17.9 ± 0.4               | 1.78 | 93.8                          |

Table 3. Comparison of the obtained values (mean ± uncertainty at the 95% level, \(\frac{\sigma}{\sqrt{n}}\)) for peach leave SRM (1547) with the reported certified values.

| Elements | Concentration (mg.kg⁻¹) | RSD% | Agreement to (SRM) values (%) |
|----------|--------------------------|------|-------------------------------|
| Al       | 310 ± 15                 | 1.42 | 90.3                          |
| As       | 0.068 ± 0.012            | 9.23 | 95.6                          |
| Cd       | 2.878 ± 0.058            | 3.08 | 93.5                          |
| Cu       | 12.22 ± 0.86             | 1.18 | 97.2                          |
| Mn       | 76.0 ± 1.2               | 0.93 | 98.4                          |
| Ni       | 2.142 ± 0.058            | 2.03 | 91.9                          |
| Se       | 0.1152 ± 0.0043          | 6.04 | 100.5                         |
| Sr       | 55.54 ± 0.50             | 3.28 | 98.7                          |
| V        | 0.568 ± 0.017            | 7.15 | 98.4                          |
| Zn       | 82.3 ± 3.9               | 1.54 | 86.3                          |

Table 4. Comparison of the obtained values (mean ± uncertainty at the 95% level, \(\frac{\sigma}{\sqrt{n}}\)) for spinach leave SRM (1570a) with the reported certified values.
Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Sb, Se, Tl, V, Zn, Th and U, 1000 mg L

The LOD and LOQ values were concentrations of 0.5, 1, 3 and 10

Table 5. Comparison of the obtained values (mean ± uncertainty at the 95% level, \( m \pm \sigma \)) for SRM 1849 – Infant/Adult Nutritional Formula with the reported certified values reported by CEM corporation. 

| Elements | Concentration (mg kg\(^{-1}\)) | Agreement to SRM values (%) |
|----------|-------------------------------|-----------------------------|
| Ca       | 4900 ± 130                    | 90.08                       |
| Cu       | 20.29 ± 0.43                  | 95.43                       |
| Fe       | 177.1 ± 3.3                   | 97.82                       |
| Mg       | 1578 ± 69                     | 92.59                       |
| Mn       | 51.00 ± 0.53                  | 95.22                       |
| Zn       | 152.3 ± 5.1                   | 97.65                       |

Table 6. ICP-MS operating conditions.

| Plasma (Ar) gas flow | 15 L min\(^{-1}\)      |
|----------------------|------------------------|
| Carrier (Ar) gas flow| 0.8–1.0 L min\(^{-1}\) |
| Collision (He or H\(_2\)) gas flow | 4.5–5.0 L min\(^{-1}\) |
| S/C temp             | 2 °C                    |
| Sampler and skimmer cons | Ni                   |
| Plasma Power         | 1500 W                  |
| Sample depth         | 6–8 min                 |
| Reflected power      | <5.0 W                  |

vessel was reduced to 3.0 mL instead of 5 to 10 mL recommended by commercial vessels. By taking into account the price of commercial vessels and ultra-pure chemicals, these vessels can be considered as more cost effective and more environmentally friendly.

Methods

The ICP-MS 7500ce (Agilent, USA) with an octopole reaction system (ORS) was used in this research. The Octopole Reaction System (ORS) is a Collision-reaction cell CRC containing an octopole ion guide in a stainless steel vessel which pressurized with a gas. Collision-reaction cell CRC is a technology to reduce or eliminate the effect of interferences coming from polyatomic species. This can be done by passing the ion beam (just before the quadrupole mass filter) through a cell that can be pressurized with a collision gas of either a reactive gas (e.g., H\(_2\), NH\(_3\), O\(_2\)) or an inert collision gas (e.g., He). Argon gas that was used throughout the experiment is of spectral purity of (99.999%). Each day prior to the beginning of the experiment, the instrument was tuned with 1.0 ppm tuning solution containing Li, Co, Y, Ce and Tl in 2.0% (v/v) HNO\(_3\) and 0.50% (v/v) HCl to cover the entire mass range to assure proper sensitivity. The settings of the instrument are reported in Table 6.

A 1000 W (Panasonic, Japan) microwave oven NN-ST651M, 32 L with inverter and Ø340 mm turntable was used throughout the experiments. An (Elga Purelab Ulq II UK) system was used to produce ultra-pure water with resistivity more than 18 MΩ.cm.

A laboratory made polytetrafluoroethylene (PTFE) vessel was designed with a relatively thicker wall compared to commercial vessels while a silicone based polymer O-ring was used as a safety valve. The suggested vessel has been patented in Iran with patent no. 71522-1390/06/26, 2011 (Rima Instrument, Iran). In this design, eight vessels were placed onto an acrylonitrile butadiene styrene (ABS) holder to keep them safe and stable. The top view of the PTFE vessel is shown in Fig. 1(a); Fig. 1(b) represents the PTFE vessels with their ABS casing. The geometrical details of the vessel and their casing are shown in Fig. 1(c,d) respectively.

The reagents used for the analysis and digestion of samples are 60% ultrapure nitric acid and 31% ultrapure hydrogen peroxide (Merck, Germany). The standard reference materials were milk powder (Infant/Adult Nutritional formula obtained from NIST U.S department) 1849a, NIST rice flour 1568b, NIST peach leave SRM 1547 and NIST spinach leave SRM 1570a.

For the calibration plot, a multi element standard Agilent with concentration of 10 mg L\(^{-1}\) for Ag, Al, As, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Sb, Se, Ti, V, Zn, Th and U, 1000 mg L\(^{-1}\) for Ca, Fe, K, Mg, Na and Sr were used. Concentrations of 50, 100, 300, and 1000 μg L\(^{-1}\) were used for five elements of Ca, Fe, K, Mg, Na and Sr, and concentrations of 0.5, 1, 3 and 10 μg L\(^{-1}\) for the rest of the elements. In Table 7 the limit of detection LOD, limit of quantification LOQ, correlation coefficient, R\(^2\), and selected modes of gas are given. The LOD and LOQ values were obtained from \( s_{BS} \) and \( 10s_{BS} \) respectively, in these equations \( s_{BS} \) is the standard deviation of blank signal and \( m \) is the slope of calibration curve.

0.10 g of each SRM sample was weighed and transferred into a PTFE vessel, then the vessels were put under the hood and 2.0 mL of ultra-pure 60% nitric acid plus 1.0 mL of ultra-pure 31% hydrogen peroxide were added into each vessel and the vessels were closed. A power to time microwave program was used for the digestion of standard reference materials. The details of power to time program used in this experiment are as shown in Fig. 2. This program was obtained by several trial and error varying the time to power program with constant amount of
In this program, the power of the microwave oven was increased stepwise, first to 167 W for 2 minutes and then it was increased to the maximum 333 W for 2 more minutes; afterwards the power was decreased to zero for one minute. This trend was repeated five times for milk powder and rice flour (SRMs) and seven times for peach and (SRM) samples (0.1 g) and digestion solution volume (3.0 mL) to gain maximum agreement with (SRM) values.

**Table 7.** ICP-MS analytical figures of merit.

| Elements | LOD (μg. L⁻¹) | LOQ (μg. L⁻¹) | Gas Mode | R² |
|----------|---------------|---------------|----------|----|
| ⁷Al      | 0.19          | 0.63          | He       | 0.9996 |
| ³⁷As     | 0.0091        | 0.0303        | He       | 1.0000 |
| ⁴⁰Ca     | 20.62         | 68.73         | He       | 0.9992 |
| ⁴⁴Cd     | 0.0035        | 0.0116        | No Gas   | 0.9999 |
| ⁴³Cr     | 0.012         | 0.040         | He       | 1.0000 |
| ⁴⁰Cu     | 0.013         | 0.043         | He       | 1.0000 |
| ⁵⁶Fe     | 0.23          | 0.77          | He       | 1.0000 |
| ⁴⁰K      | 3.41          | 11.37         | He       | 1.0000 |
| ⁴⁰Mg     | 0.16          | 0.53          | He       | 1.0000 |
| ⁵⁵Mn     | 0.064         | 0.213         | He       | 1.0000 |
| ⁵²Mo     | 0.016         | 0.053         | He       | 1.0000 |
| ⁴⁰Na     | 0.35          | 1.16          | He       | 1.0000 |
| ⁶⁰Ni     | 0.046         | 0.153         | He       | 1.0000 |
| ⁴⁰Pb     | 0.0092        | 0.0306        | No Gas   | 0.9994 |
| ³⁴Se     | 0.0029        | 0.0097        | H₂       | 1.0000 |
| ⁶⁰Sr     | 0.041         | 0.1367        | He       | 0.9998 |
| ⁵¹V      | 0.178         | 0.593         | He       | 1.0000 |
| ⁶⁴Zn     | 0.038         | 0.127         | He       | 0.9975 |

Figure 1. (a) Top view of a PTFE vessel, (b) PTFE vessels with their ABS casing, (c) vessel geometry, (d) (ABS) holder geometry.
spinach leaves (SRMs). After digestion, the vessels were put under the hood to cool down. The contents were filtered through a 0.45 μm PTFE, if necessary (only peach and spinach leaves SRMs), and then transferred into a 50 ml polypropylene volumetric flask and diluted with ultrapure water to the marked level. The diluted samples were stored in polyethylene vials until the time of analysis by ICP-MS.

For cleaning, 3.0 mL of analytical grade nitric acid was poured into each vessel. After closing the vessels, a similar program with that used for digestion was applied to wash the vessels for 15 minutes. This vessel armed with silicone O-ring to release any over pressure that may occur because of applying higher power of microwave radiation or longer time of applying radiation. However, in this power to time microwave program, it was tried to apply power and time of radiation to digest different standard reference materials in a way to prevent the overpressure condition. This was archived by several experimental cycles.

It has been reported that, when water is heated in a closed vessel microwave, the internal pressure is lower than that in closed vessels heated by conventional methods. This is due to the vessel materials and the heating mechanism. Since the closed vessel microwave and their outer casing are microwave transparent, they remain relatively cool during the heating process. Therefore, the vessel’s wall becomes cooler and causes water molecules to remove from vapor phase. Moreover, it was reported that simultaneous application of air flow outside the digestion vessel can improve the efficiency of digestion in the microwave oven. Consequently, this enhancement in condensation rate causes a decrease in internal pressures at higher temperatures. This is similar to a reflux action, which explains the better digestion. With the same conclusion, the cooling and heating period, shown in Fig. 2, simulates the reflux action and causes very good agreement to (SRM) values. These vessels armed with silicone O-ring to release any over pressure that may occur because of applying higher power of microwave radiation or longer time of applying radiation.

Figure 2. Power to time microwave program used for digestion of different SRMs.

Figure 3. (a) Silicone rubber O-ring, (b and c) silicone rubber O-ring after deformation.
The PTFE vessels used in this research were equipped with a silicon-based polymer with high tensile strength and temperature resistance properties. In the case of enhancement of pressure at high temperature, this silicone rubber deforms to release the excess pressure. Figure 3(a) shows the silicone rubber O-ring before deformation and its temperature resistance properties. In the case of enhancement of pressure at high temperature, this silicone rubber will not cause the activation of safety system of this vessel, therefore, better recoveries can be observed in this investigation.

References
1. Aras, N. K. & Ataman, O. Y. Trace element analysis of food and diet. Royal Society of Chemistry 7, 10.1039/9781847552495 (2006).
2. Demirel, S., Tüzen, M., Saracoglu, S. & Soyak, M. Evaluation of various digestion procedures for trace element contents of some food materials. Journal of Hazardous Materials 152, 1020–1026, doi:10.1016/j.jhazmat.2007.07.077 (2008).
3. Nemati, K., Abu Bakar, N. K., Abas, M. R. B., Sobhanzadeh, E. & Low, K. H. Comparative study on open system digestion and microwave assisted digestion methods for metal determination in shrimp sludge compost. Journal of Hazardous Materials 182, 453–459, doi:10.1016/j.jhazmat.2010.06.053 (2010).
4. Bizzi, C. a. et al. Effect of simultaneous cooling on microwave-assisted wet digestion of biological samples with diluted nitric acid and O2 pressure. Analytica chimica acta 837, 16–22, doi:10.1016/j.aca.2014.05.051 (2014).
5. Matusiewicz, H. in Microwave-Assisted Sample Preparation for Trace Element Analysis (ed Érico Marlon de Moraes Flores) 77–98, http://dx.doi.org/10.1016/B978-0-444-59420-4-00004-0 (2014).
6. Sucharova, J. & Suchara, I. Determination of 36 elements in plant reference materials with different Si contents by inductively coupled plasma mass spectrometry: comparison of microwave digestions assisted by three types of digestion mixtures. Anal Chim Acta 576, 163–176, doi:10.1016/j.aca.2006.06.004 (2006).
7. Bermejo-Barrera, P., Moreira-Páveo, a., Muñiz-Naveiro, O., Gómez-Fernández, a. M. J. & Bermejo-Barrera, a. Optimization of a microwave-pseudo-digestion procedure by experimental designs for the determination of trace elements in seafood products by atomic absorption spectrometry. Spectrochimica Acta Part B: Atomic Spectroscopy 55, 1351–1371, doi:10.1016/S0584-8547(00)00239-1 (2000).
8. Chandrasekaran, S., Ramanathan, S. & Basak, T. Microwave food processing—A review. Food Research International 52, 243–261, doi:10.1016/j.foodres.2013.02.033 (2013).
9. Makedonski, L.; Peycheva, K. & Stancheva, M. Determination of some heavy metal of selected black sea fish species. Food Control, doi:10.1016/j.foodcont.2015.08.024 (2015).
10. Sorbo, A., Túrco, A. C., Di Gregorio, M. & Ciarami, L. Development and validation of an analytical method for the determination of cadmium, lead and lead content in powdered infant formula by means of quadrupole Inductively Coupled Plasma Mass Spectrometry. Food Control 44, 159–165, doi:10.1016/j.foodcont.2014.03.049 (2014).
11. Tüzen, M., Sesi, E. & Soyak, M. Trace element levels of mushroom species from East Black Sea region of Turkey. Food Control 18, 806–810, doi:10.1016/j.foodcont.2006.04.003 (2007).
12. Zain, S. M., Behkami, S., Bakirdere, S. & Koki, I. B. Milk authentication and discrimination via metal content clustering – A case of comparing milk from Malaysia and selected countries of the world. Food Control 66, 306–314, doi:10.1016/j.foodcont.2016.02.015 (2016).
13. Abu-Santia, M., Mohlbauer, A., Morris, J. S. & Kortyohann, S. Wet ashing of some biological samples in a microwave oven. Analytical Chemistry 47, 1475–1477, doi:10.1021/ac60358a103 (1975).
14. Kingston, H. M. & Jassie, L. Microwave-Energy for Acid Decomposition at Elevated-Temperatures and Pressures Using Biological and Botanical Samples. Analytical Chemistry 58, 2534–2541, doi:10.1021/ac00125a038 (1986).
15. Araújo, G. C. L., Gonzalez, M. H., Ferreira, A. G., Nogueira, A. R. A. & Nóbrega, J. A. Effect of acid concentration on closed-vessel microwave-assisted digestion of plant materials. Spectrochimica Acta Part B: Atomic Spectroscopy 57, 2121–2132, doi: http://dx.doi.org/10.1016/S0584-8547(02)00164-7 (2002).
16. Bizzi, C. a. et al. Improvement of microwave-assisted digestion of milk powder with diluted nitric acid using oxygen as auxiliary reagent. Spectrochimica Acta Part B: Atomic Spectroscopy 66, 394–398, doi: http://dx.doi.org/10.1016/j.sab.2011.04.013 (2011).
17. Costa, L. M., Silva, F. V., Gouveia, S. T., Nogueira, A. R. A. & Nóbrega, J. A. Focused microwave-assisted acid digestion of osh: an evaluation of the residual carbon content. Spectrochimica Acta Part B: Atomic Spectroscopy 56, 1981–1985, doi: http://dx.doi.org/10.1016/j.sab.2007.05.003 (1991).
18. Matos, W. O. et al. Partial microwave-assisted wet digestion of animal tissue using a baby-bottle sterilizer for analyte determination by inductively coupled plasma optical emission spectrometry. Spectrochimica Acta Part B: Atomic Spectroscopy 64, 615–618, doi: http://dx.doi.org/10.1016/j.sab.2009.05.005 (2009).
19. Pucilota, R. S. et al. Simultaneous determination of bromine and iodine in milk powder for adult and infant nutrition by plasma based techniques after digestion using microwave-induced combustion. Spectrochimica Acta Part B: Atomic Spectroscopy 107, 86–92, doi: http://dx.doi.org/10.1016/j.sab.2015.02.007 (2015).
20. Saavedra, Y., González, A., Fernández, P. & Blanco, J. A simple optimized microwave digestion method for multielement monitoring in mussel samples. Spectrochimica Acta Part B: Atomic Spectroscopy 59, 533–541, doi: http://dx.doi.org/10.1016/j.sab.2003.12.024 (2004).
21. Yang, K. X. & Swami, K. Determination of metals in marine species by microwave digestion and inductively coupled plasma mass spectrometry analysis. Spectrochimica Acta Part B: Atomic Spectroscopy 62, 1177–1181, doi: http://dx.doi.org/10.1016/j.sab.2007.07.007 (2007).
22. Yang, K. X., Swami, K. & Hussain, I. Determination of trace metals in atmospheric aerosols with a heavy matrix of cellulose by microwave digestion-inductively coupled plasma mass spectrometry. Spectrochimica Acta Part B: Atomic Spectroscopy 57, 73–84, doi: http://dx.doi.org/10.1016/S0584-8547(01)00354-8 (2002).
23. Afridi, H. L., Täipärä, P. N., Kazi, T. G. & Brabazon, D. Assessment of toxic elements in the samples of different cigarettes and their effect on the essential elemental status in the biological samples of Irish hypertensive consumers. J Hum Hypertens 29, 309–315, doi:10.1038/jhh.2014.87 (2013).
24. Punshon, T. et al. Placental arsenic concentrations in relation to both maternal and infant biomarkers of exposure in a US cohort. J Expo Sci Environ Epidemiol 25, 599–603, doi:10.1038/jes.2015.16 (2015).
25. Bettinelli, M., Baroni, U. & Pastorelli, N. Microwave oven sample dissolution for the analysis of environmental and biological materials. Analytica Chimica Acta 225, 159–174, doi:10.1016/S0003-2670(00)84604-8 (1999).
26. Richter, C. R., Link, D. & Kingston, H. M. S. Peer Reviewed: Microwave-Enhanced Chemistry. Analytical Chemistry 73, 30 A-37 A, doi:10.1021/ac0123781 (2001).
27. Polkowska-Motruk, H., Danko, R., Dybczyński, R., Koster-Ammerlaan, A. & Bođe, P. Effect of acid digestion method on cobalt determination in plant materials. Spectrochimica Acta Part A 408, 89–95, doi:10.1016/j.sab.2010.08.007 (2010).
28. CEM corporation. Preparation of Mixed Food Samples for Trace Metals Analysis Using the CEM MARS 6 and Discover SP-D Systems, http://cem.com/media/contenttype/media/literature/516_ApNote_MARS6_DiscSPD_MixedFoods.pdf (2016) (Date of access:08/10/2016).
**Acknowledgements**
The authors wish to express their gratitude to the research council of Marvdasht Islamic Azad University for their financial support and University of Malaya, UMRG grant no. RG308-14AFR.

**Author Contributions**
Mehrdad Gholami designed and manufactured the device and with the help of Shima Behkami carried out the experiment. Shima Behkami, Sharifuddin Md. Zain and Sezgin Bakirdere carried out the data analysis and final corrections. All authors reviewed the manuscript.

**Additional Information**

**Competing financial interests:** The authors declare no competing financial interests.

**How to cite this article:** Gholami, M. *et al.* A simple design for microwave assisted digestion vessel with low reagent consumption suitable for food and environmental samples. *Sci. Rep.* **6**, 37186; doi: 10.1038/srep37186 (2016).

**Publisher's note:** Springer Nature remains neutral with regard to jurisdictional claims in published maps and institutional affiliations.

*This work is licensed under a Creative Commons Attribution 4.0 International License. The images or other third party material in this article are included in the article’s Creative Commons license, unless indicated otherwise in the credit line; if the material is not included under the Creative Commons license, users will need to obtain permission from the license holder to reproduce the material. To view a copy of this license, visit [http://creativecommons.org/licenses/by/4.0/](http://creativecommons.org/licenses/by/4.0/)*

© The Author(s) 2016