PERFORMANCE COMPARISON OF MICROWAVE CAVITIES USED FOR EXTRACTION OPERATIONS

GIACINTO D'AMORE[1], GIUSEPPE D'AURIA[2], ALDO ROMANO[2] AND FRANCESCO MARRA[1]

Abstract:

In the agri-food industry world, billions of tons of waste are produced every year. These represent both a direct loss (due to the failure to exploit their potential value, and their nutritional and energy content) and indirect, due to their necessary treatment and/or disposal. Some of substances contained in the wastes, of potential high value, can be recovered by means of extraction. Conventional extraction processes involve the use of solvents, which end up requiring an additional process of separation from the solute identified as the desired product. In recent years, extraction techniques have been proposed without the use of solvents.

In this work, a performance comparison of microwave cavities used for extraction operations is presented. Particularly, the work addressed two cavities, both working at 2.45 GHz. A calorimetric analysis performed by following the heating rate and temperature evolution in rack of 25 beakers filled with 25 ml of water, coupled with the solution of the heat transfer balance in the system, allowed to build the spatial distribution of the electromagnetic power dissipated as heat in each of the beakers. Fluid-dynamics aspects related to the recovery of the vapour phase produced during the extraction were also analyzed, with particular emphasis to the mean residence time of the vapour fraction in the extraction chamber as a function of its configuration.

Keywords: microwaves, extraction, process intensification

Introduction

In the newest approach to chemical industrial processes there is a claim that some basic principles have to accomplished to respect a more general trend of clean sustainable development (CHEMAT ET AL., 2012), such as energy saving, renewable sources, reduced unit operation, robust processes, that can be synthetized as process intensification. Actually, extraction is one of the most difficult operations to be performed according to the recommendations above, since the tremendous impact that typically has on the environment, for its large consumption of energy, of water in conventional plants and of raw materials due to is relatively low yield.

[1] Dipartimento di Ingegneria Industriale – Università degli studi di Salerno, Fisciano SA Italy
[2] Dipartimento di Farmacia – Università degli studi di Salerno, Fisciano SA Italy

Nevertheless, the industrial interest for extraction is growing, as new fields are now open to the bioactive compounds obtained, such as nutraceutics and cosmeceutics areas. However, this even potentially enormous market pretends purity grades far higher than chemical industry is used to accept.

To this purpose, new methods for the extraction of bioactive compounds from vegetable matrices have been proposed in the last years, all of them trying to get rid of all the drawbacks presented by either expensive, low efficiency or toxic solvents. Use of microwaves appears to overcome all the difficulties above, as the effect of their interactions with vegetable matrices is to generate an endogenous water vapor that, in turn, forms a hetero-azeotrope (BERK, 2013) with essential oils and other bioactive molecules, allowing the extraction without solvents. The method seems to offer a great potential, but still requires as much accuracy in the design and implementation of the necessary equipment.

There is evidence that the use of microwaves reduces process times, improving yields or even making possible extraction processes otherwise difficult to achieve (ROBINSON ET AL., 2010). However the variety of possible systems (Single mode, Multimode, Traveling Wave Tubes, Cavity, Open Applicator *et caetera*), more than solving, makes the choice more difficult due to the presence of drawbacks in each system. Two are the crucial points of an extraction by microwaves: i) the field distribution, that has to be as much homogeneous as possible not to cause inhomogeneities in the sample undergoing the irradiation, and the relative microwaves power control, as it drives the whole process; ii) the design of the extraction chamber, with special reference to the internal fluid dynamics of all the extracted compounds. The literature presents paper aimed at describing the field in cavities by fairly complex mathematical evaluations (MENDEZ-SANCHEZ ET AL., 2003, CURET ET AL., 2008), sometimes not in perfect agreement with the experimental results or related to model systems far from the real apparatuses.

This work proposes a simple but effective calorimetric method that allows to describe how the specific power - generated by the interaction of the electromagnetic field with the load matrix- is distributed in a set load placed in a microwave cavity; given two different cavities, the presented method allows to highlight differences the two cavities have in terms of microwave field distribution.

Materials and Methods

The microwave systems. In this work. the choice of microwave extraction was taken taking into account the presence of water in the vegetable matrices of interest, so as to operate in a solvent-free mode. For the purposes of optimizing the plant for the extraction process, two microwave cavities were used and compared, with different modes of operation: a commercial type, with the magnetron working with duty-cycles, defined by an operating phase at maximum power followed by one at magnetron off, and a modified mode, with the magnetron constantly working during the whole process time. The following points were analyzed: i) the behavior of electromagnetic waves within the two cavities; ii) the advantage, if any, deriving from a specially developed microwaves generator with a linearly variable control of the delivered power; iii) the choice of geometry for the extraction vessel.

The first microwave cavity used (Figure 1), indicated with W, initially without modifications, was a commercial-type one, 800 W (size L = 31.7 cm; H = 21 cm; W = 31.5 cm), equipped with a reflecting wave stirrer that homogenizes the electromagnetic field as much as possible; the second one Figure 2), called L (size L = 25.8 cm; H = 19 cm; W = 30 cm), was modified by replacing the original magnetron with a 900 W one, whereas the plate rotation mechanism was removed to allow housing in safety of the extraction vessels. Moreover, a generator for a linearly variable power supply was added.

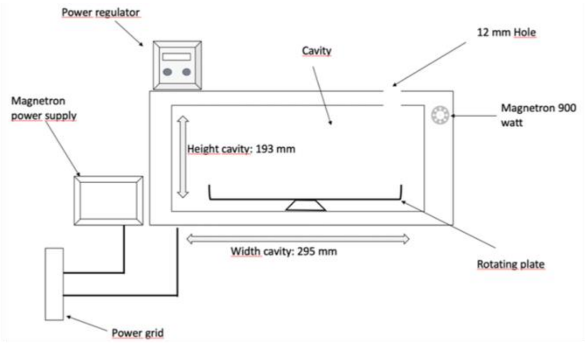


Figure 1. Plant sketch. Cavity W

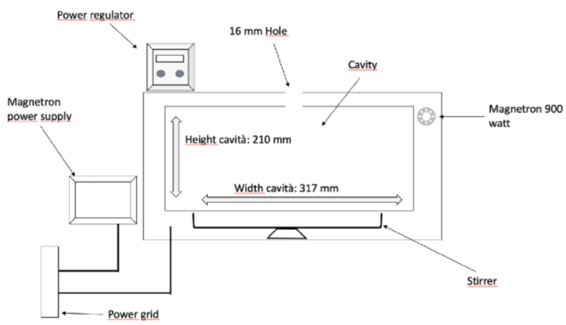


Figure 2. Plant sketch. Cavity L

<u>The grid</u>. To study and verify the differences between the electro-magnetic fields of the two above described instruments a simple but effective calorimetric method that allows to describe how the specific power - generated by the interaction of the electromagnetic field with the load matrix- is distributed in a set load placed in a microwave cavity. To this purpose, on a grid of 25.5 cm x 21.5 cm, 25 intersection points were defined, lying on 5 columns and 5 rows. On the points were placed beakers, filled with 25 ml each of water. The tests were carried out by individually monitoring each column through the use of optical fibers for temperature measurement. The same modus operandi was followed for both ovens. The calorimetric tests were carried out on both the commercial series and the modified cavities, thus with and without the duty cycle function.

The temperature measurements were carried out with a temperature sensing instrument of the company FISO, composed by a signal conditioner, a data processing program and optical fiber probes with a detectable temperature range from -40 ° C to 300 ° C, a length of 2 m, a diameter of 1 mm and a measurement error of +/- 1 ° C.

Heat balance

Under the hypothesis that the conductive and convective contributions to the heat transfer are negligible, the balance of heat allows the calculation of specific power generated by the microwaves in the water samples, Q_{mw}:

$$\rho\ Cp\ \frac{\partial T}{\partial t} = Q_{mw} \qquad (1)$$

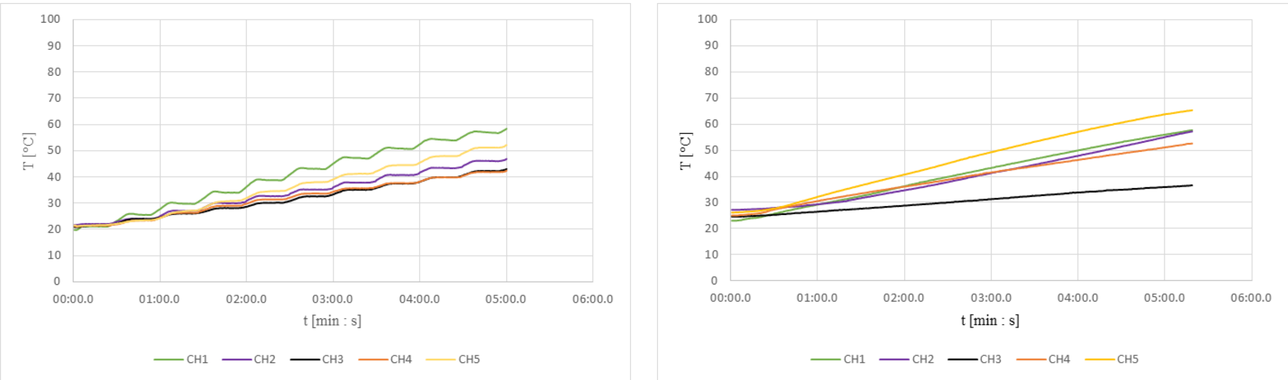
where ρ is the mass density, Cp is the heat capacity, T is the temperature, t is the time.

Using eq.(1), the specific power generated by the microwaves in the water samples was estimated and analyzed as function of time and space.

Results

Results are shown in terms of temperature and specific power evolution and distribution in the two considered microwave cavities.

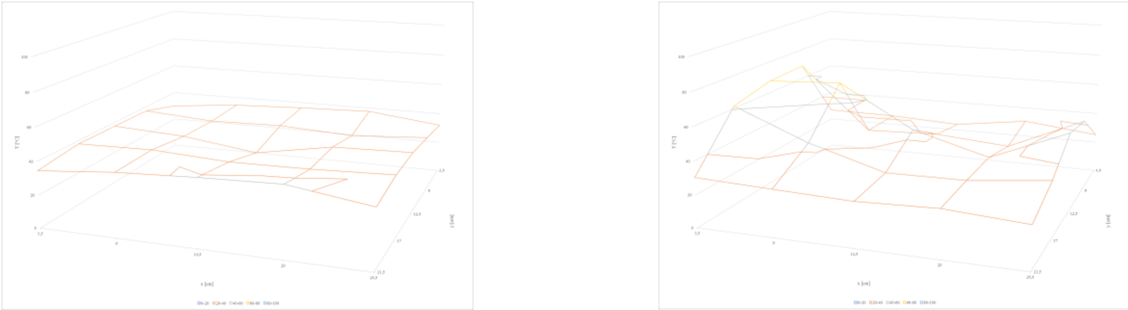
The temperature-time evolution in the five beakers placed on the central column of the samples' grid in cavity W and cavity L are shown in figures 3.a and 3.b respectively.



a) b)

Figure 3: Temperature-time evolution in the five beakers placed on the central column of the samples' grid in cavity W (a) and cavity L (b). Channel 1: back; Channel 5: front.

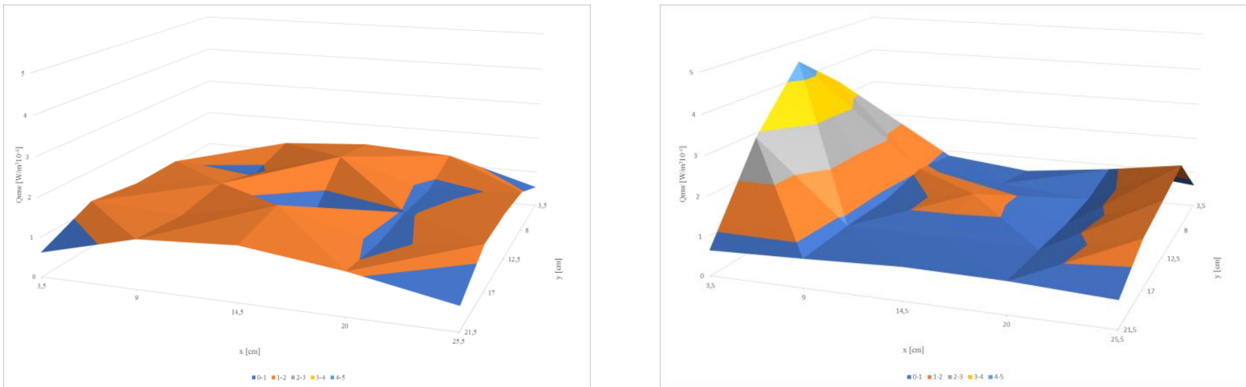
In figures 4.a and 4.b, the temperature distribution in cavity W and cavity L after 120 seconds of exposition to 350 W microwave power are shown.



a) b)

Figure 4: Temperature distribution after 120 seconds of heating at mean output power of 350 W in cavity W (a) and cavity L (b).

The specific power distribution (computed as in eq.(1)) in cavity W and cavity L during the exposition to an average microwave power of 350 W are shown in figures 5.a and 5.b respectively.



a) b)

Figure 5: Specific microwave power distribution during exposure to an average microwave power of 350 W in cavity W (a) and cavity L (b).

Discussion

The performances of the two investigated cavities changed in terms of temperature-time evolution and in temperature and specific power distribution.

Samples in cavity W were subjected to an intermittent heating, as proven by the alternate series of temperature ramps and plateaus along the time line (figure 3.a), whereas the samples in cavity L exhibited a very linear trend of temperature versus time, position by position. The linearity of temperature vs time was also a proof that the hypothesis on the basis of the heat balance equation was correct, since both conductive and convective contribution to heat transfer in the water samples look being negligible.

Temperature distribution in samples hold in cavity W resulted more even and uniform than in those hold in the case of cavity L, which resulted hot in some areas and much colder in others.

Particularly, the beakers hold in cavity L, in the position farer from the waveguide port exhibited the higher temperature values.

The uneven distribution of sample temperatures in cavity L was reflected by the specific microwave power distribution, which appeared – for this cavity - non uniform and with a point at which the specific power overcame 4e5 W/m^3, while in cavity W the specific microwave power distribution was uniform with values bounded between 1e5 and 2e5 W/m^3.

It appeared evident that the magnetic stirrer was more effective in determining the evenness of heating than the continuous microwave power driver used in cavity L.

It is clear that in case of a specific power distribution like in cavity L, a sample hold in the area of higher microwave energy impact would suffer because of the over exposition to the waves, with consequent over heating or even arching or burning in some cases, like emphasized in figure 6. At same time, the samples treated in cavity L presented zones of the grid where there is risk of under heating (or under processing), with then scarce exploitation of the microwave energy and inefficient processing.

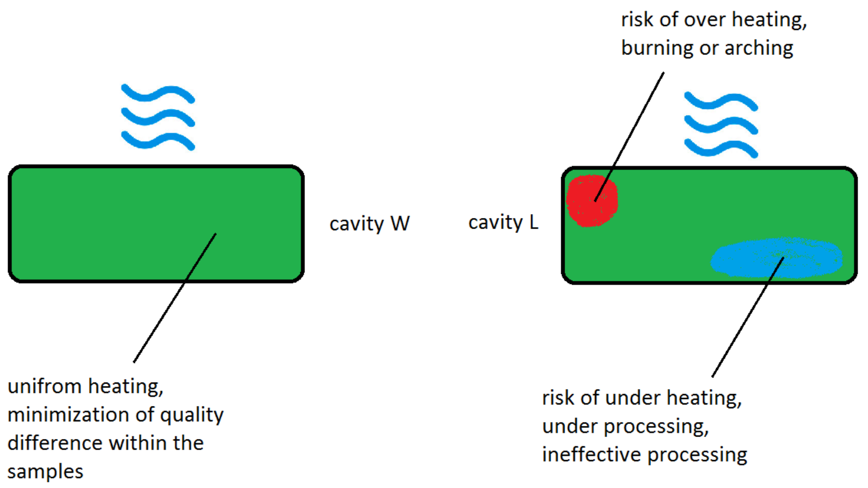


Figure 6: Sketch of overall microwave power distribution in the two considered cavities, W and L.

Conclusions

A simple but effective calorimetric method has been proposed to study the different temperature-time evolution and the spatial temperature and specific microwave power distribution in an array of samples hold in two microwave cavities different one each other by the microwaves distribution systems (cavity W was equipped with a magnetic stirrer) and the microwave power supplier (cavity L was equipped with a continuous microwave power driver. Cavity W, even if run with a classical on-off duty cycle, thanks to the presence of a magnetic stirrer just in front of the waveguide port, allowed to obtain uniform distribution of temperature and specific microwave power.

Further work is needed to explore how the distribution of the specific microwave power changes with the planar position of the samples' array and, eventually, what benefit can derive from the application of a continuous microwave power supplier to the cavity W, equipped with the magnetic stirrer.

Acknowledgments

This work was done in the framework of Microexativa project, run at University of Salerno, Italy.

"Progetto cofinanziato dall'Unione Europea, dallo Stato Italiano e dalla Regione Campania, nell'ambito del POR Campania FESR 2014-2020" Microexativa - Estrazione senza solvente, mediante microonde, di principi attivi per la cosmetica e la nutraceutica da matrici di Cannabis Sativa"

References

Berk, Z., (2013) *Food Process Engineering and Technology,* Chapt. 13, 346-348, Elsevier, ISBN 978-0-12-373660-4

Chemat, F., Maryline, O., Vian, A., Cravotto G. (2012) *Green Extraction of Natural Products: Concept and Principles* International Journal of Molecular Sciences 13(7), 8615-8627

Curet, S., Rouaud, O., Boillereaux, L. (2008) *Microwave tempering and heating in a single-mode cavity:Numerical and experimental investigations* Chemical Engineering and Processing 47 1656–1665

Mendez-Sanchez, R.A., Kuhl, U., Barth, M., Lewenkopf, C. H., Stockmann, H.J. (2003) *Distribution of Reflection Coefficients in Absorbing Chaotic Microwave Cavities,* Physical Review Letters 91, (17) 174102 1-4

Robinson, J., Kingman, S., Irvine, D., Licence, P., Smith, A., Dimitrakis, G., Obermayer , D., Kappe, O. (2010) *Understanding microwave heating effects in single mode type cavities—theory and experiment*, Phys. Chem. Chem. Phys., , 12, 4750–4758