CALORIMETRIC DETERMINATION OF MICROWAVE ENERGY ABSORPTION IN - RESONANT OR MULTIMOD APPLICATORS IN A CONTINUOUS-FLOW REACTOR

Daniela GHIMPEȚEANU[1*], Vasile LAVRIC[1], Ioan CĂLINESCU[1], Mariana PĂTRAȘCU[2]
[1]University POLITEHNICA of Bucharest, Faculty of Applied Chemistry and Materials Science, Bioresources and Polymer Science Department, 1-7 Gh. Polizu street., 011061, Bucharest, Romania
[2]Chemspeed SRL, 2 Gara de Nord street., 010856, Bucharest, Romania

Abstract

Microwaves (MWs) cover a broad spectrum of applications, all having in common the MWs capacity of deploying energy "in situ", throughout the interactions matter-electromagnetic field, overcoming the losses implied when the same amount of energy should be transferred through one or several interfaces. When it comes to process intensification, the key is the efficiency of the deployed energy usage by the process.

In this work, we present the performances of a new applicator based upon two concepts: resonance and focus of the electromagnetic field (EM) on the target. At resonance frequency, the cavity stores the MWs energy – therefore, no MWs will be reflected back into the wave guide. The Q factor, proportional to the ratio of energy stored in the resonator and the energy dissipated per cycle, is a measure of the frequency selectivity of the resonator. When the applicator is at resonant frequency, the Q factor is very high. When the dimensions of the applicator are not in resonance with the MWs frequency, the Q factor drops drastically, since the reflected power starts having high values. The reflection coefficient, S11, witnesses how much of the delivered power is reflected back to the wave guide – the lower the value is, the smaller the fraction of power reflected back. The focusing capacity of the applicator is measured with the liquid absorbed power yield, η_{PL}, defined as the ration between the total power dissipated in the liquid phase and the total power introduced in the applicator. Closer to one the values of the yield, higher the applicator capacity to focus the MWs energy on the target.

In the case of MWs with a frequency of 2.45 GHz and TE_{10} mode, the smallest resonant cavity is a cube with the side equal to 86.525 mm. Unfortunately, when placing a load in the resonant cavity and attached a wave guide to it, the interactions matter-electromagnetic field will change the latter, increasing the energy reflected back. Starting from the aforementioned side, the dimensions of the new applicator were searched such that the liquid absorbed power yield to be maximum – the corresponding new side is 145.26 mm.

This new applicator was tested, both in laboratory and in COMSOL Multiphysics® modeling software, for several liquids with very different loss tangent magnitudes and temperature behavior, namely water, ethylene glycol, cyclohexane, acetic acid and 2-propanol, flowing through a special type of reactor, vertically coiled. The MWs generator is a solid-state Miniflow, with a maximum power of 200 W and a special integrated circuit to measure the direct and reflected power. A conventional multi-mode applicator with a special adaptor ensuring the transition from the coaxial guide to WR340 guide, thus permitting the usage of the same solid-state generator, was employed as reference against the new applicator, doing the

---

[*] Corresponding author: Daniela Ghimpeteanu, Email address: ghimpeteanu_daniela@yahoo.com

same experimental and simulation work.

The parameters used to compare the performances of both applicators are:

- *the specific power absorbed by the liquid phase (SAR);*
- *the liquid absorbed power yield;*
- *the Q factors;*
- *the reflection coefficient.*

Key words: Microwave, New Applicator, Comsol Multiphisics Modeling Software, Dielectric Parameters

1. Introduction

Microwave heating technology has been used in many areas as a means of selective, volumetric and instantaneous heating. In the process, the electrical energy is first converted into microwave energy, which is then absorbed by a dielectric medium and converted into thermal energy. A better understanding of the efficiency of these methods is needed by quantitative measurement to optimize the microwave heating process and to use it more efficiently. Research reported so far has divided the process of microwave heating in two stages, measuring in detail the efficiency of energy use and studying the effects for different factors. The result of the experiment can vary greatly depending on the position of the sample inside the cavity, the type of heating medium, the microwave output power and the geometric parameters of the heating medium, as well as the ratio between the volume of the sample and the volume of the applicator.

Microwave heating technology uses the most commonly used electromagnetic waves being 915 MHz and 2450 MHz to heat specific materials. Because it is a selective, volumetric and very rapid heating, this method has been widely used in drying, organic synthesis, pyrolysis of biomass and waste, processing of polymeric materials, removal of pollutants from gas or water and many others [3].

So far, there are many simulated data and very few experimental results. A common problem when using microwaves is the variation of the dielectric properties, the shape and dimensions of the sample that is heated.

In several experimental tests it was shown that in the multimodal applicator, the efficiency of the microwaves varies according to the frequency, the dielectric properties, the position of the reactor inside the cavity as well as its geometry [2].

In the work written by Monzo-Cabrera, J. Pedreno-Molina, J.L. Toledo, T, shows that for several dielectric samples, the efficiency varies according to the distance from the magnetron, following a nonlinear curve, noticeable differences of the energy efficiency values between the best and the weakest positions are reported.

The microwave power absorbed by the sample can be determined by the temperature difference between the initial and final moments, when the sample has been applied a certain microwave treatment.

It is possible to define the energetic behavior of a microwave applicator by the efficiency parameters, which represents the relationship between the incident power and that reflected in the power supply guide. When a single power port is used, the energy efficiency and the reflection coefficient are related to the following equation. Where: η - energy efficiency, S11 - the reflection coefficient for the power port.

$$\eta = 1 - |S_{11}|^2 \tag{1}$$

On the other hand, the microwave power absorbed by the sample may be related to the increase in temperature difference, as explained in Equation 2. Where: Pabs (W) is the microwave power absorbed by the sample, m_{sample} (Kg) is the mass of the sample, Cp (J/ Kg · C) is the specific heat, Δt (° C) temperature difference and, t, is the irradiation time[2].

$$\mathrm{Pabs} = \mathrm{m_{sample}} \cdot \mathrm{Cp} \cdot \Delta \mathrm{t/t} \tag{2}$$

The efficiency of the conversion from microwave energy (Qmicrowave) to effective heat (Qef) can be calculated using the following equation [3].

$$\eta = Q\,ef / Qmicrowave \tag{3}$$

2. Comparison of dielectric parameters of common organic solvents and water at 2.45 GHz

The most important characteristic of a solvent under MW irradiation conditions are the dielectric constant (Ɛ’), the dielectric loss factor (Ɛ‘’), and the dissipation factor (tan ƍ). The dielectric constant (Ɛ’) depends on the frequency of the MW radiation and temperature. Dielectric loss (Ɛ‘’) represents the quantity of input MW energy that is lost to the sample by being dissipated as heat. The ratio of the dielectric loss to the dielectric constant is an important factor that determines the heating rate of the microwaves. The ability of a substance to convert electromagnetic energy into heat is determined by the dissipation factor (tan ƍ = Ɛ‘’/ Ɛ‘). Therefore, when the dielectric factors of the sample are compared, it is necessary that comparison be made at some fixed temperature [1].

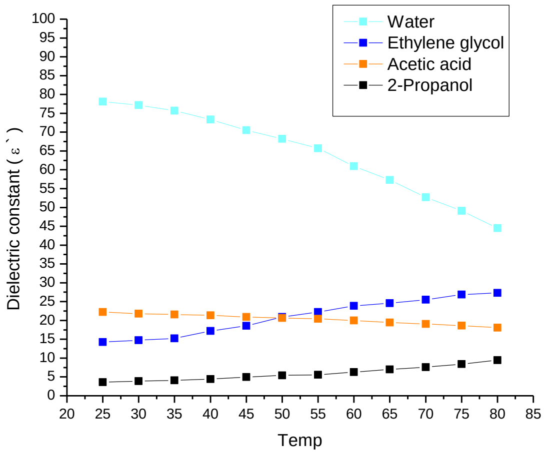


Fig. 1 Dielectric constant (Ɛ’)

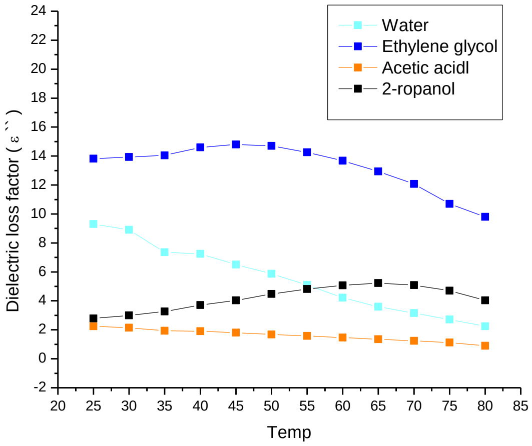


Fig. 2 Dielectric loss factor (Ɛ‘’)

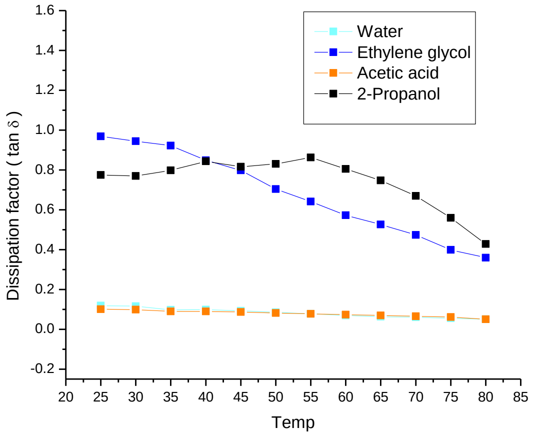
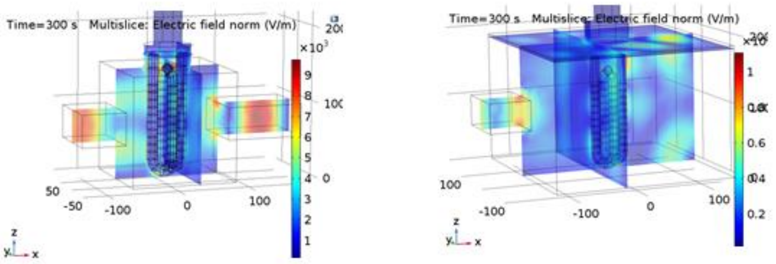


Fig. 3 Dissipation factor (tan ɗ = Ɛ‘’/ Ɛ‘)

3. Comsol Multiphsics® modeling software

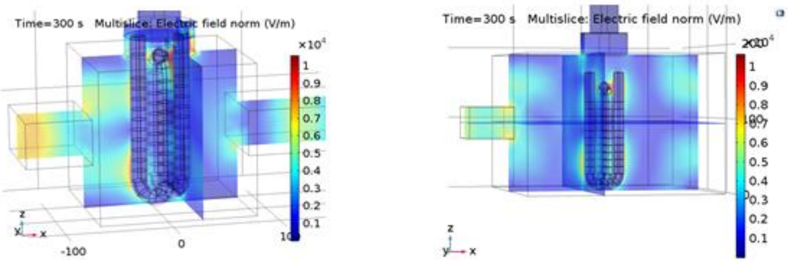
Comparative study of the performances of resonant and multimodal applicators using a vertical spiral reactor in dynamic regime. The influence of the dielectric properties of the liquid phase.

Regardless of the fluid used, the inlet flow rate in the reactor is 50 ml / min, the inlet temperature is 25°C, and the power introduced in the applicator is 50 W, with a simulated time of 300 s.



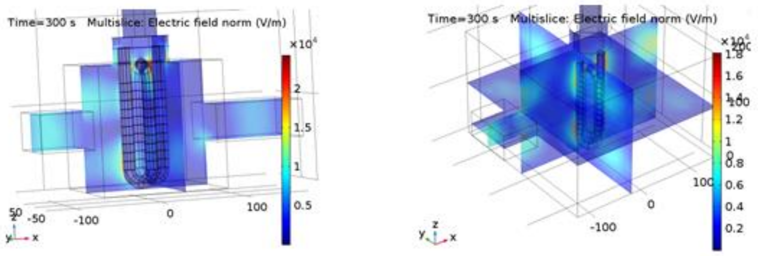
Resonant, water

Multimodal, water



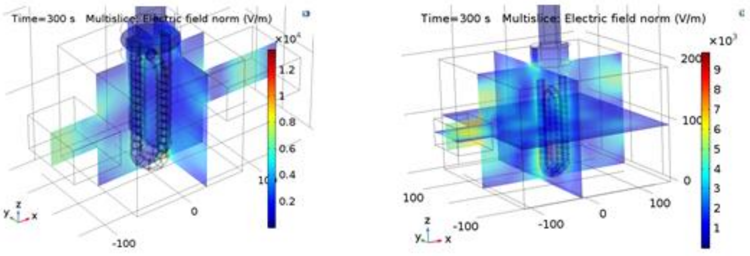
Resonant, ethylene glycol

Multimodal, ethylene glycol



Resonant, acetic acid

Multimodal, acetic acid

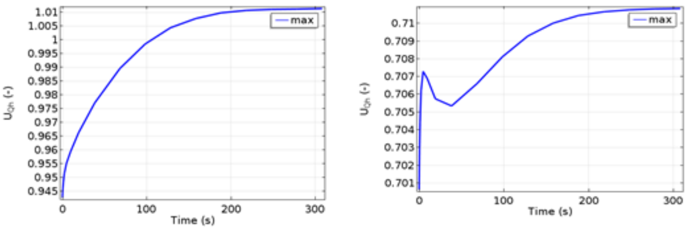


Resonant, 2-propanol

Multimodal, 2-propanol

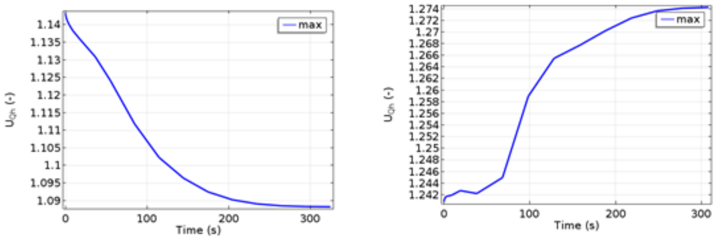
Fig. 4 Spatial distribution of the electric field for distilled water, ethylene glycol, acetic acid, 2-propanol, for the resonant applicator, respectively, the multimodal laboratory applicator.

The uniformity index of the thermal dissipation, UQh, which represents the ratio between the average temperature and its standard deviation - a high value indicates a small standard deviation, compared to the average, therefore, a more uniform distribution.



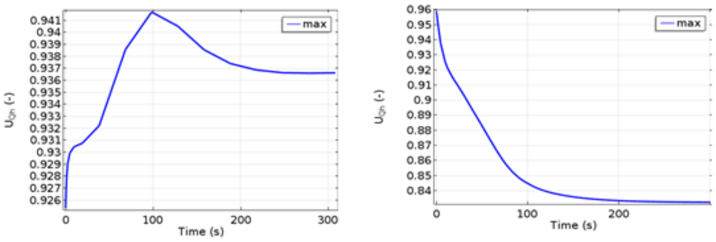
Resonant, water

Multimodal, water



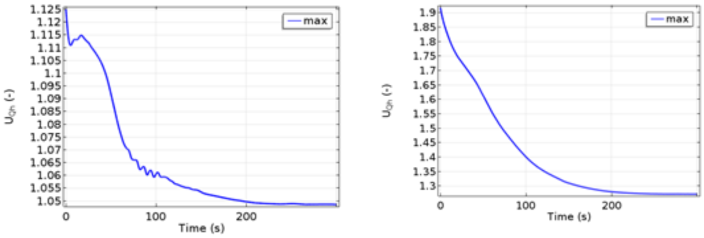
Resonant, ethylene glycol

Multimodal, ethylene glycol



Resonant, acetic acid

Multimodal, acetic acid

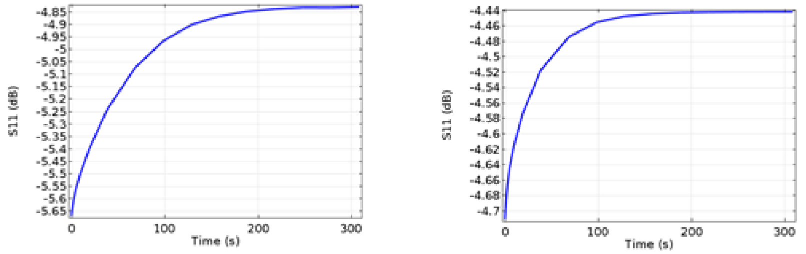


Resonant, 2-propanol

Multimodal, 2-propanol

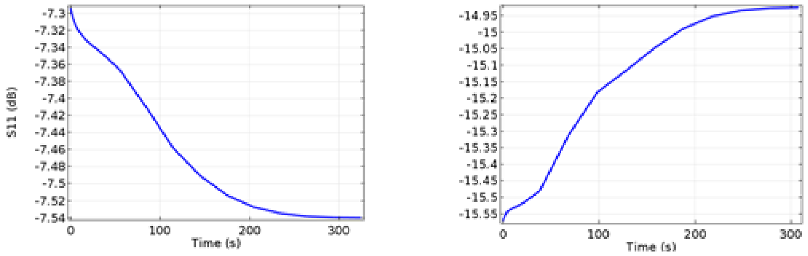
Fig. 5 Temporal variation of the uniformity index of thermal dissipation in the entire liquid phase, for distiled water, ethylene glycol, acetic acid, 2-propanol, in single-mode resonant and multimodal applicator

Another important coefficient, which shows how much of the energy introduced into the cavity is reflected back, is the parameter S11dB = 20 · log10 (| S11 |). The lower its values, the lower the reflected power. A better behavior of the multimodal applicator is observed, the parameter S11dB having lower values than those of the resonant applicator.



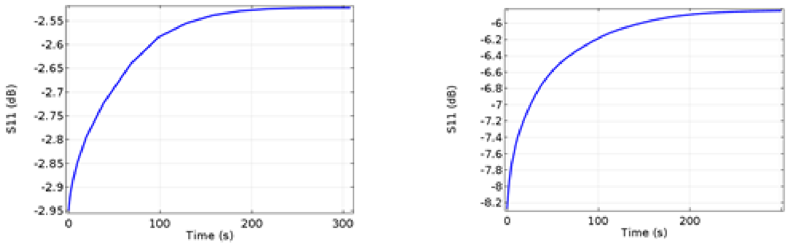
Resonant, water

Multimodal, water



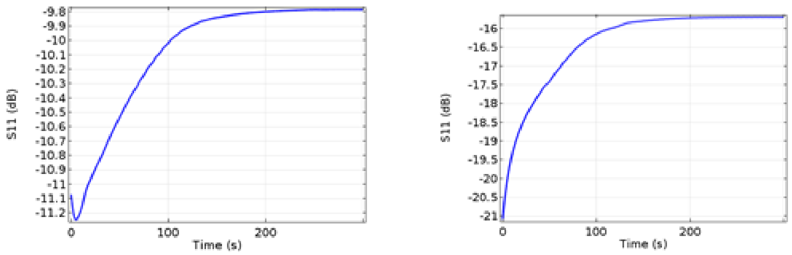
Resonant, ethylene glycol

Multimodal, ethylene glycol



Resonant, acetic acid

Multimodal, acetic acid

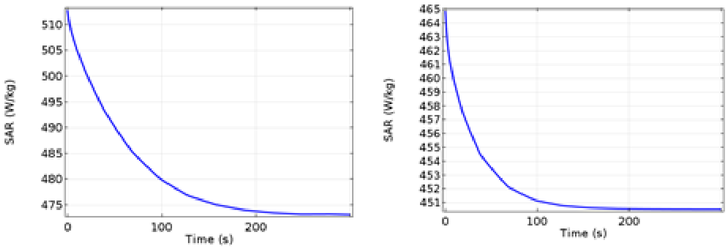


Resonant, 2-propanol

Multimodal, 2-propanol

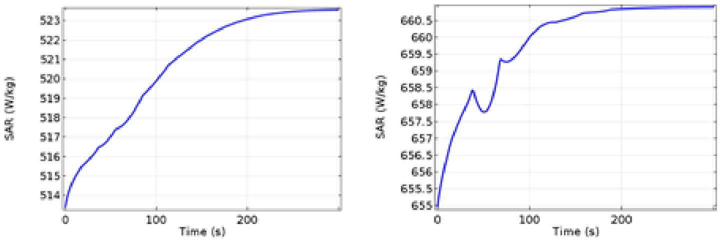
Fig. 6 Temporal variation of parameter S11dB for distilled water, ethylene glycol, acetic acid, 2-propanol, in single-mode resonant and multimodal applicators.

Another parameter of interest, for performance comparison, is the specific power absorbed, SAR (W / kg). Due to the increase of the liquid phase temperature in time and space, the temporal profile of the SAR changes decreasing, to the value corresponding to the thermodynamic balance.



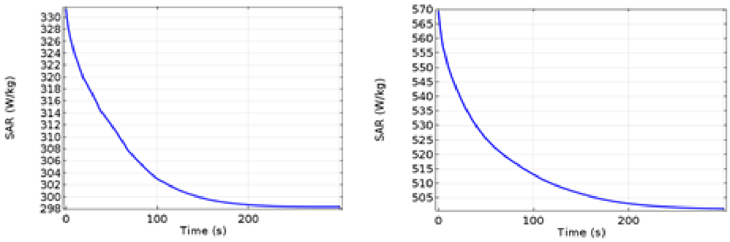
Resonant, water

Multimodal, water



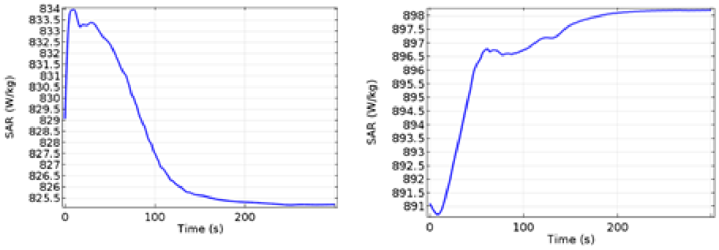
Resonant, ethylene glycol

Multimodal, ethylene glycol



Resonant, acetic acid

Multimodal, acetic acid



Resonant, 2-propanol

Multimodal, 2-propanol

Fig. 7 The temporal variation of SAR for distilled water, ethylene glycol, acetic acid, 2-propanol, in single-mode resonant and multimodal applicator

4. Setup and method

We have carried out activities that allow the experimental investigation of a new microwave applicator (the ability to take microwave energy from liquids with different dielectric properties) in order to optimize the enzymatic pretreatment of lignocellulosic biomass. For this purpose, an experimental installation was carried out by means of which a liquid with the preset temperature and constant flow is pumped through a vertical spiral type reactor which is irradiated with microwaves. The absorbed power by the liquid in the reactor was determined calorimetrically. As a microwave source, the Miniflow solid-state generator was used. Different temperatures were used, at which the dielectric properties of the studied liquid are known and experiments were conducted in the cavity itself and in a multimodal applicator specially adapted to be able to be supplied with microwave energy from the solid-state generator.

In this experiment the infusion of microwaves in a flow system and a reactor in the form of a a vertical spiral, is monitored, by monitoring the temperature at the exit of the reactor. They will be performed in two different equipments, depending on how the microwave energy is applied to the sample, namely: the multimodal system and the single-mode resonant system.

The aim is to establish a working mode for the single mode and multimodal applicator by testing with 4 liquids with different properties. The liquids used are distilled water, ethylene glycol, glacial acetic acid, isopropanol.

Initially, the thermostat and the peristaltic pump are switched on to homogenize the fluid in the system. After a few minutes, the temperature monitoring system and MiniFlow are switched on and left for two minutes to record without microwaves (Initial Temp. 1). Then the microwaves are started at a power around 50 W and left on until the temperature remains constant (Final Temp). After the microwaves are turned off, the temperature is monitored until it returns to its initial value (Initial Temp 2). The temperature is measured in thermostat and at the exit of the reactor with fiber optic, but also with thermocouple. For each temperature set, 2 determinations are made, from 25 ° C to 75 ° C, from 10 to 10 degrees. In each determination, the initial and final temperature are noted when the microwaves are turned off. The power generated by MiniFlow was set to 50 W for all experiments and at 25 ° C more power was tested. For water and ethylene glycol the powers of 25, 50, 75, 100 W were tested and for glacial acetic acid and cyclohexane were tested at 50, 100 and 180 W.

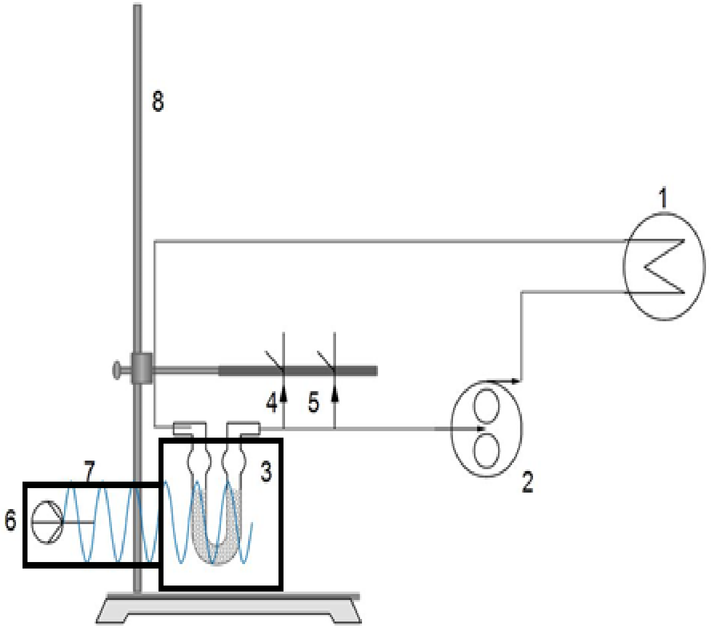


Fig. 8 Experimental set up
1 - Thermostat, 2 - Peristaltic Pump, 3 - Reactor, 4 - Fiber Optics, 5 - Thermocouple,
6 - MiniFlow 200SS, 7 - Microwave Guide WR340

The fluid flow is passed through the peristaltic pump (2) through the thermostat (1), and the liquid used reaching a desired temperature. From the thermostat the liquid enters in a vertical spiral reactor (3). In the reactor the sample is exposed to microwaves at a power of 50 W, generated by MiniFlow (6). The temperature is monitored, for each experiment, using fiber optics (4), but also with a thermocouple (5).

5. Results and discussion

As it can be seen in the graphs below (Fig.9), for distilled water, we have a considerable difference between the multimodal and the resonant applicators.

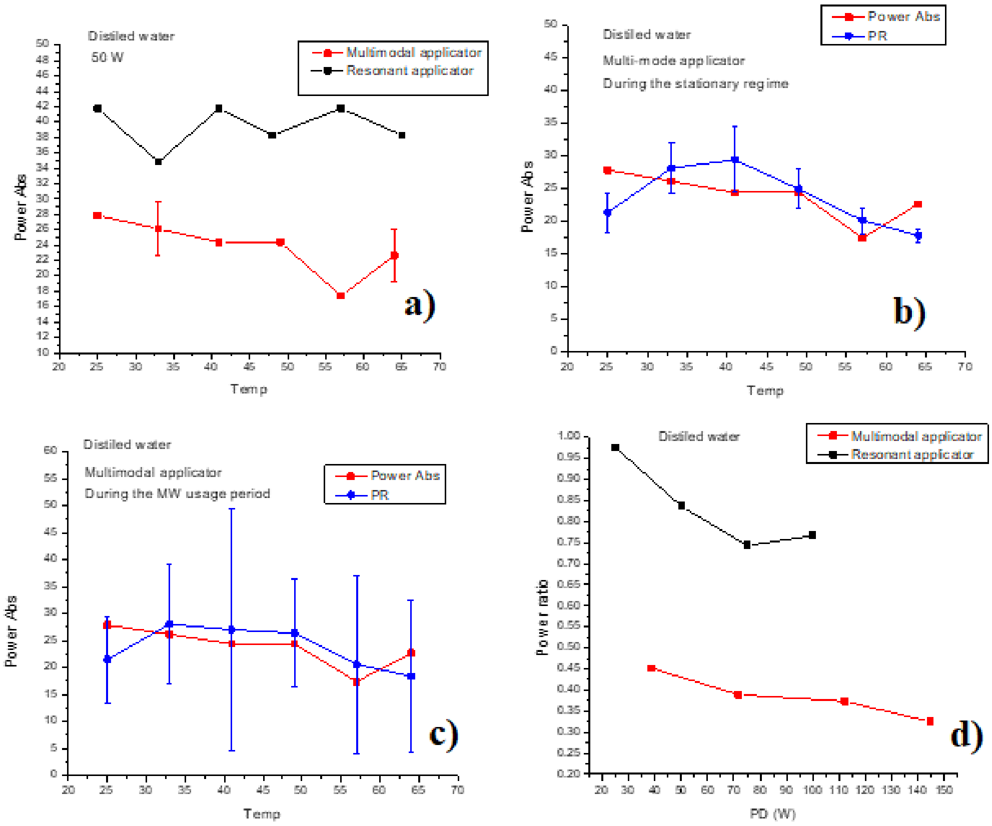


Fig. 9. Results for resonant and multi-mode applicators using distilled reference liquid distilled water

In these graphs we have the values of the experiments for the resonant and multi-mode applicators using as reference liquid distilled water. In the image a) the power absorbed can be observed depending on the temperature; In the image b) the power absorbed according to the temperature can be observed for the multimodal applicator (in the experiments with distilled water only in the multimodal applicator we had reflected power) but also the reflected power (PR) during the stationary regime period for each experiment with the minimum values and maximums for the respective period; In the image c) the power absorbed according to the temperature can be observed for the multimodal applicator, but during the whole heating period (300-400 seconds); and in image d) the differences between the power ratio and the direct power for the two applicators are represented.

In Fig. 10, we have presented the values of the experiments for the resonant and multimodal applicators using as reference liquid ethylene glycol.

In Fig.11 and 12 the results for acetic acid as well as for 2-propanol we have reflected power in the multimodal applicator but also in the resonant applicator.

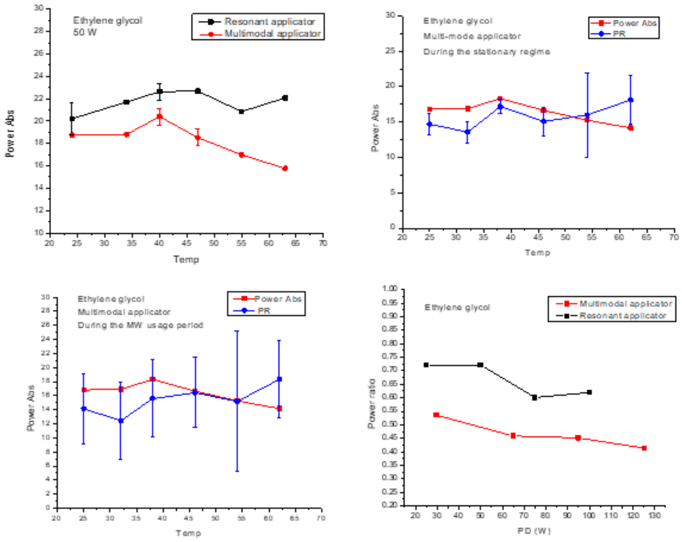


Fig. 10. Results for resonant and multi-mode applicators using distilled reference liquid ethylene glycol

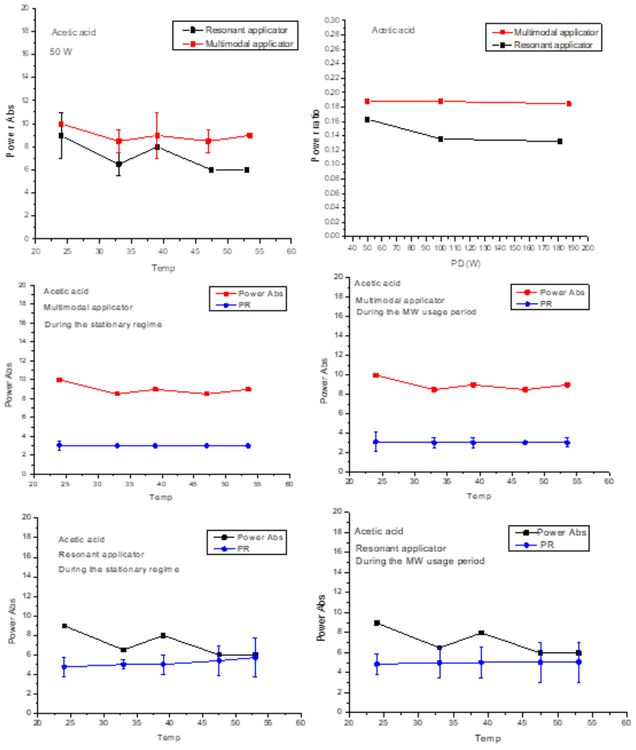


Fig. 11. Results for resonant and multi-mode applicators using distilled reference liquid acetic acid

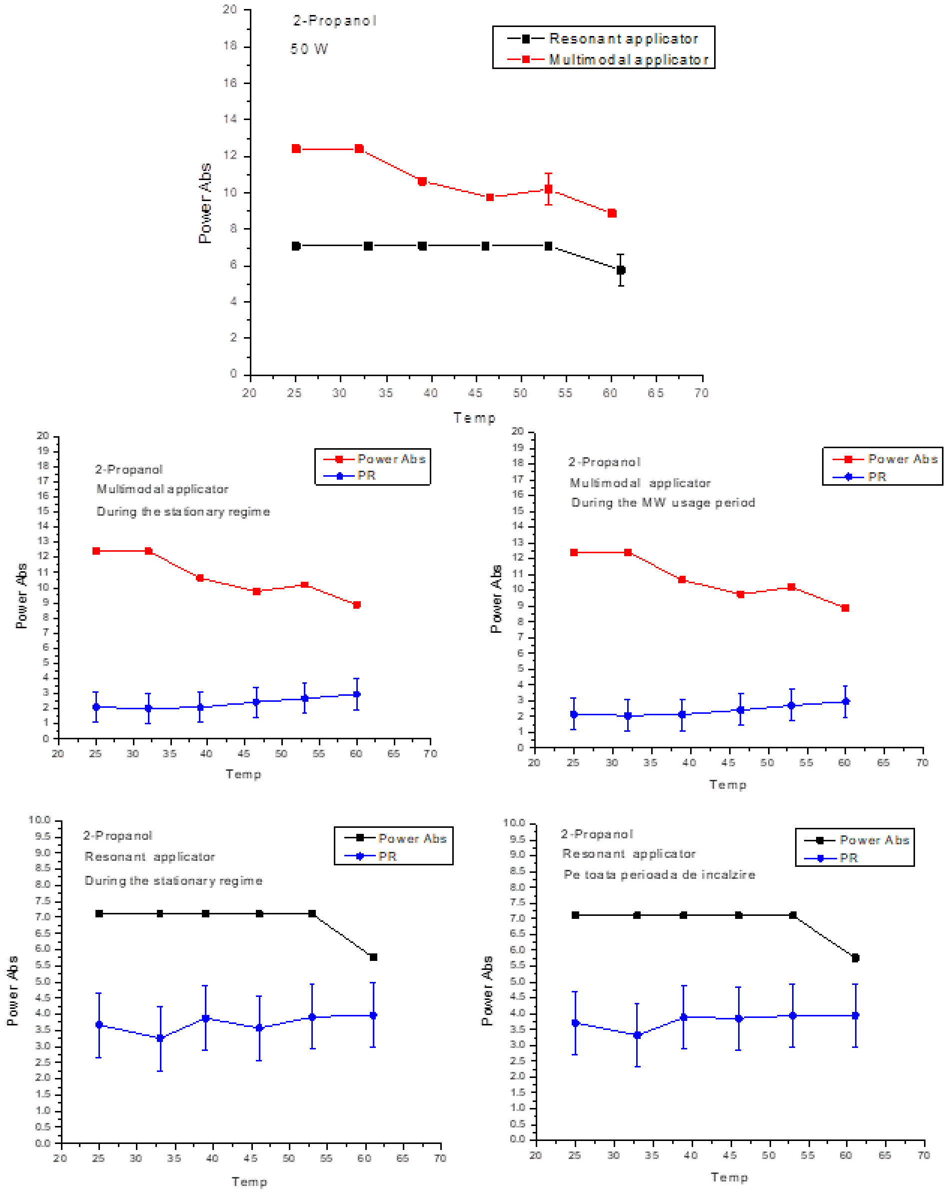


Fig. 12 Results for resonant and multi-mode applicators using distilled reference liquid 2-propanol

6. Conclusions

In the case of modeling in Comsol as well as experimental, we have the same results except for ethylene glycol, where the results are very close for the 2 applicators.

A considerable difference can be observed when using distilled water as the reference liquid between the two applicators. The resonant applicator is superior to the multimodal one.

For acetic acid and 2 propanol we have reflected power for both types of applicators. We can reduce the reflected power to a minimum by using Stub Automatic Tuner.

These differences between the 4 liquids tested in the 2 applicators are due to the dielectric factors.

Acknowledgment
The authors acknowledge the financial support received from the Competitiveness Operational Programme 2014 - 2020, Action 1.1.4: Attracting high-level personnel from abroad in order to enhance the RD capacity, ID project: P_37_471, MY SMIS 105145, Ultrasonic/Microwave nonconventional techniques as new tools for nonchemical and chemical processes, financed by contract: 47/05.09.2016.

REFERENCES

[1] Horikoski S., Matsuzaki S., Mitani T., Serpone N., Microwave frequency effects on dielectric properties of some common solvents and on microwave-assisted syntheses: 2-Allylphenol and the C_{12}-C_2-C_{12} Gemini surfactant, *Radiation Physics and Chemistry*, 81,(2012),1885-1895.
[2] Monzo-Cabrera J., Pedreno-Molina J.L., Toledo T., Feedback control procedure for energy efficiency optimization of microwave-heating ovens, *Elsevier,* 42, (2009), 1257-1262.
[3] Wang W., Zhao C., Sun J., Quantitative measurement of energy utilization effciency and study of influence factor in typical microwave heating process, *Elsevier,* 87, (2015), 678-68.