Is Microwave-Assisted Chemistry Scalable? A Tutorial Guide for Chemists

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1. Introduction

In my 20 years of using microwaves, I have been a chemist and researcher, and became a microwave chemist. My first microwave equipment was a domestic kitchen oven… Since then, I have been an end-user, an equipment designer and manufacturer, a teacher, and a business woman selling microwave equipment and technology to different industries around the world. I always get excited (and sometimes frustrated) when talking about microwaves, and especially in the context of chemistry and plasma applications. In this article, I would like to share with you my experience and vision, mostly as a guide for chemists entering the field.

I always believed that for the microwaves to succeed in industry, the industrial microwave manufacturer ought to design and provide the researcher with good, reliable equipment, and equally, with all information and training to ensure correct usage of the equipment and the safety of the users.

Since the introduction of microwaves as a tool for heating, a variety of successful industrial applications have emerged. Food processing, rubber heating and wood treatment are just a few examples. Why isn't the chemical industry included yet in this success? At least three different reasons come to mind, namely (a) publications, (b) the equipment manufacturers, and (c) chemistry researchers and their general understanding of the microwave hardware. These factors are further elaborated below:

1.1. Published work

Thirty years or so after Gedye's first use of kitchen microwave-oven for enhancing chemical reactions, some progress has been made. However, yet 90% of the published papers still report work done in a kitchen oven, more or less 'dedicated to chemists', which cannot be considered valid for scale-up.

Microwave-related research is mainly published in scientific journals reviewed by non-microwave reviewers. Similarly, books on microwave synthesis are mostly authored by chemists. Consequently, most publications in the field do not seem to focus on the microwave hardware as a discipline by itself.

1.2. Microwave equipment manufacturers

Most dedicated microwave reactors for chemists are made by equipment manufacturers using 'cooking with microwaves' techniques. These are more expensive, with fancy touch screens and user-friendly interfaces allowing for different degrees of sophistication with respect to process control, database capabilities, safety features, and different vessel designs. However, with no information on the most important microwave parameters – namely the forward and reflected power, and hence the absorbed power vs. time – these microwaves are just heating devices for fast reactions and high activation energy. Most of the dedicated microwave laboratory equipment are built using relatively high-power magnetrons. These are applied by researchers for processing small samples, and sometimes this combination leads to too high microwave-power densities, which are not scalable in industrial reactors. As there is no possibility to closely monitor and control the reaction as it proceeds, it is very often proposed to work in a pressurized reactor since the solvents and/or reagents can be heated at temperatures higher than their boiling point.

Generally speaking, dedicated microwave laboratory equipment manufacturers are not the same as industrial equipment manufacturers. This also impedes the scaling-up feasibility. In addition, due to size and price considerations, the 2.45-GHz frequency is mainly preferred for dedicated laboratory equipment whereas industrial equipment is more likely designed to use 915 MHz.

1.3. *Chemistry researchers*

Due to high prices of dedicated microwave reactors, chemists still prefer using kitchen ovens to perform reactions. Too often, the chemist is not interested in what is inside this ‘black box’, and the most monitored parameter is the temperature, or perhaps the pressure, without associating them with the absorbed microwave power and power density available from the electromagnetic (EM) field.

Reaction scale-up encompasses several aspects of chemistry, chemical engineering and fluid mechanics. Issues arising from kinetics, thermodynamics and hydro-dynamics, affect the selection of the best type of reactor, its internal design, and its operating regime. Now we need to add EM waves, temperature-dependent dielectric properties, power density, arcing, etc. Developing safe, robust, and scalable processes, calls for a close interdisciplinary collaboration between researchers, equipment manufacturers, and end-users. However, ground reality and publications reveal that, barring a few exceptions, both researchers and industrial-equipment manufacturers fail to put this principle into practice.

So, what do the non-microwave chemists need to know in order to enable reliable and scalable experimentation? Apart from knowing the way microwaves interact with matter, as often discussed and published, the chemists must also be familiar with the basics of the microwave hardware.

2. Microwave hardware

A typical microwave setup (based on either kitchen oven, laboratory equipment or industrial units) consists of four main components[1]: the microwave generator, the matching or tuning element, transmission lines, and the applicator within which the product to be heated is placed. These three main components can be installed together in one metal enclosure (similar to a kitchen oven) or lined-up in a given order as illustrated in Fig. 1.

2.1. *Magnetron-based microwave generators*

A typical magnetron-based microwave generator consists of a high-voltage (HV) power supply (L/C transformer or switched-mode converter) and a magnetron. The latter is a microwave auto-oscillator in a vacuum tube consisting of an external cylindrical corrugated anode usually made out of copper, and a heated axial cathode, made out of tungsten impregnated with emissive material. Heating the cathode to ~ 1,750 ^{0}C results in the emission of electrons, which generate the electric field; a permanent magnet for power up to 6 kW, or an electromagnet for higher power ratings, applies a crossed magnetic field. Within the magnetron, the conversion of the electrons’ kinetic energy into useful microwave energy also generates heat due to the impact of the electrons with the anode. The dissipation of this heat is done by air cooling in the case of kitchen and low-power industrial magnetrons (up to 2 kW); industrial magnetrons above 2 kW are cooled by water and air.

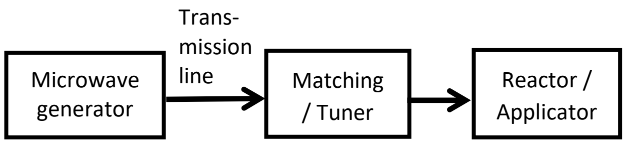


Figure 1. A general setup of microwave equipment. The generator may consist of a magnetron with a HV power-supply, or a solid-state (transistor) generator. The transmission line could be a coaxial cable or a waveguide (for low and high power, respectively). The reactor is a single or multi-mode cavity, or a continuous-flow tunnel.

The frequency of the magnetron, its maximum power and mains electrical efficiency (conversion of electrical into microwave power) are mainly governed by the magnetron geometry, distance between the anode and the cathode, and the anode geometry and size. The electrical efficiency of 2.45 GHz magnetrons exceeds 70 % while that of 915 MHz/896 MHz may reach ~ 90 %.

Most industrial magnetrons use either transformer power-supplies or switched-mode converters, which are more advanced. The half-period transformer circuit used in low-cost laboratory and kitchen ovens just turns the oven ON and OFF at full power hence the average power delivered to the load is determined by the duty cycle, as shown for instance in Fig. 2. In this example, the duty cycle is 40 % and the average power is P_{avg} = 0.4 x 800 = 320 W.

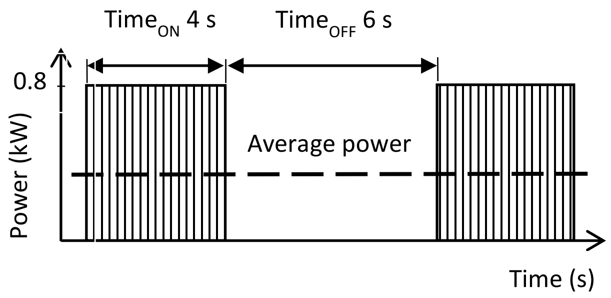


Figure 2. An example of duty-cycle control of the average output power of a magnetron (square pulses are shown for the sake of representation only; in reality the microwave pulses have a non-square shape)

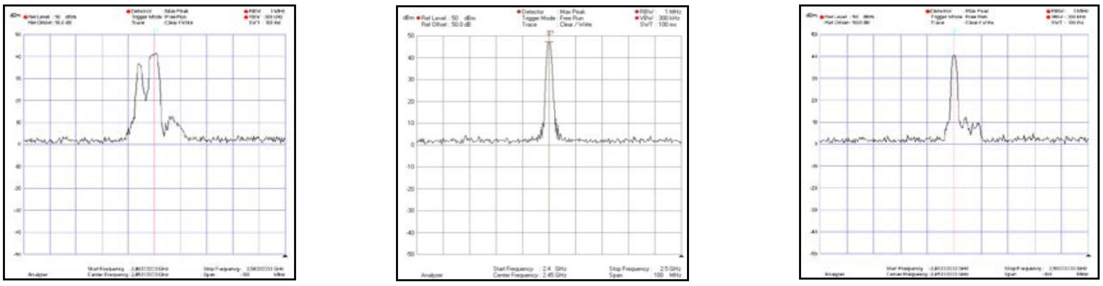
In most cases, the average power calculated (e.g. the 320-W above) is not entirely absorbed by the sample. The power absorption by the sample depends on many parameters, such as the sample size, the geometry of the vessel in which the sample is placed, its position inside the equipment, the efficiency of the multimode cavity, etc. The user does not have any direct indication from the equipment as regards to the power dissipated inside the sample. The calculation of the power dissipated in the cavity and absorbed by the sample is mainly given by the difference between the incident and reflected power, i.e. the power demanded by the user and delivered by the magnetron, the efficiency of the cavity and the reflected power. However, none of these parameters are indicated in such basic instruments.

In addition, the relatively high power sent by the instrument at the very beginning (see Fig. 2) makes it impossible to monitor the reaction, especially when working with small samples. If the sample happens to be located in a high absorption position inside the equipment, blasting 800 W even for a few seconds may mean the reaction is finished before having the time to measure anything; the reaction times could be less than the time required by the measuring instruments to reach the actual value of any parameter.

The magnetron is a consumable and must be replaced once the emission of the electrons by the cathode ceases or weakens. Industrial magnetrons differ from kitchen magnetrons; industrial magnetrons are designed to operate in harsher conditions, e.g. much higher power levels, longer running time than kitchen magnetrons, and sometimes several microwave On/Off cycles during a day. The magnetron's lifetime is expressed in total number of microwave emitting hours. For example, if a microwave generator operates 24 h/day, i.e. ~8500 h/year, and taking the average lifetime of ~7000 h, it follows that the magnetron must be replaced approximately three times in two years; however, if the microwave generator operates ~8 h/day, the magnetron replacement will be only once in two years. This calculation should make it easier to understand why a kitchen magnetron 'never dies'… An average operating time of 5 to 10 minutes a day for 365 days means 30 to 60 hours per year, which can result in many years of operation. For easier interpretation, the emission, losses and cooling of magnetrons can be simplified as the equivalent phenomena happening in an incandescent light bulb.

To auto-oscillate, the magnetron needs minimum voltage and current; CW magnetrons are known to produce an acceptable spectrum quality above ~10 % of their nominal (rated) power (e.g. a typical 1.2 kW magnetron operates at its best beween ~0.12 to 1.2 kW). Low power levels lead to instability of the magnetron's emission and operation.

While in operation, the frequency of the magnetron shifts due to metal dilatation (cathode and anode); the frequency variation depends highly on the temperature, and also on the output microwave power (see Figs. 3 a, b). These variations may create problems especially when the magnetron is used to deliver power to high quality-factor cavities, for example when the frequency shifts beyond the resonant frequency of the cavity in which the microwave treatment takes place. To insure stable operation, the cooling of the magnetron is very important. Due to environmental air temperature changes, air cooled magnetrons installed in most commercial laboratory microwave equipment are more prone to unreliable operation than the water cooled ones. In the case of water cooling, the water for cooling the magnetron ought to be provided from a closed circuit chiller with controlled temperature and pressure.



(a) P = 280 W (b) P = 1,200 W (c) P = 200 W (solid state)

Figure 3 (a, b) Spectral emission of a 2.45-GHz, 1200-W magnetron (YJ1540-3, water cooled) at various power levels; and **(c)** the spectral emission of a solid-state generator for comparison. The center frequency is 2,450 MHZ in all three spectrograms, and the horizontal and vertical scales are 10 MHz/Div and 10 dB/Div, respectively.

In addition to the environment and the type of the power supply and power control, the frequency stability and the spectrum of a magnetron are highly dependent on the load (i.e. the sample to be heated and the cavity where the sample is placed). Badly designed cavities and small, poorly absorbing (low-loss) samples can create high levels of reflected power, which affects the operation characteristics. From a hardware point of view, most manufacturers of industrial microwave generators offer built-in circulators or isolators to protect the magnetron against reflected power. Units to measure the reflected power are also provided. However, most of the manufacturers of the commercially available laboratory equipment do not install isolators or reflected-power detectors in their systems. Due to all above considerations, the user must pay special attention when testing at low-power settings, with small samples and low absorbing samples.

The microwave absorbance of most load materials varies with changes in temperature, phase and chemical composition. This results in a gradual or rapid shift in the power absorbed by the load and therefore, for a fixed forward power, an increase in reflected power, risk of arcing, changes in the magnetron's frequency and temperature, which are common problems in obtaining reproducible results.

2.2. Solid-state microwave generators

Recently, the solid-state microwave generator has become available for chemists. I consider it a great tool for every single microwave laboratory. From a view point of laboratory testing, there are great features available with this generator. Unlike magnetron-based generators, which are using vacuum tubes, the solid-state microwave generators have the electrons inside semiconductor materials, i.e. transistor. Due to the fact that solid-state generators act like amplifiers, they are able to produce an excellent frequency spectrum over the full range of power from the very first watts. Other advantages include frequency and phase variability and controllability, low input-voltage requirements, compactness, reliability, and better compatibility with other electronic circuitry. In addition, several manufacturers of such generators have built in isolators and forward/reflected power measurements making the setup a lot easier for the chemist. The possibility to continuously control and adjust the frequency over the S-band (particularly in the range 2.4–2.5 GHz) is also used by equipment manufacturers as an automatic tuning feature to minimize the reflected power as the reaction proceeds. The user can run the equipment from a few watts, in increments of 1-2 W.

In practice, the maximum power presently delivered by a single transistor is ~250 W at 2.45 GHz, and ~600 W at 915 MHz, which ought to be more than enough power to carry out basic research. Manufacturers of such generators[2] may also merge multiple power-transitor blocks within a single generator to increase its microwave power output. However, there are other concerns, especially when transmitting high power via a coaxial cable which has certain losses (depending on the dimenions and filling of the cable, and the type of metal connectors used at both ends). Slightly inferior with respect to magnetrons, practical solid-state microwave generators yield <60 % electrical efficiencies; they can be air cooled for low-power laboratory usage or air-and-water cooled for high power uses.

2.3. Microwave applicators

Metallic enclosures into which the samples to be heated and launched microwave signals are introduced, namely microwave cavities, can be classified in two main categories, single mode and multimode. Once launched into the cavity, the electromagnetic wave will undergo multiple reflections. The superposition of the incident and reflected waves gives rise to a standing-wave pattern, which is well defined in space for single mode cavities and more complex for multimode structures.

There are two parameters of importance in this regard for the chemist. One is the cavity quality factor, determined by the amount of microwave power dissipated or absorbed by the cavity itself when operated empty. The other is the resonance frequency.

Single-mode resonant cavities are structures with well-defined distribution of the electric field; their size is determined by the radiation wavelength. Such cavities come in a number of shapes and geometries, although rectangular cavities based on sections of waveguides and cylindrical cavities based on metal tubes are the most common. The single-mode cavity may establish a higher electric-field strength than a multimode applicator, hence it is more useful for treatment of low-loss dielectrics and small samples. As the properties of the processed materials change, the coupling characteristics (measured by the reflected power) shall be continuously monitored and adjusted. Poor coupling not only results in a low heating efficiency but can also lead to damage of the equipment (e.g. by arcing).

Chemists are encouraged to work in such structures for basic research, and small or poorly absorbing samples. The minimum microwave components required for such structure are shown in Fig. 1. It includes a microwave generator (preferably a solid-state or CW magnetron with switch-mode power supply) with clear indication of the incident and reflected microwave power (from the generator and load, respectively). Means for impedance matching (either manual or auto-tuned) are also essential. These could be capacitive or inductive impedance elements in the form of obstacles inserted into the waveguide (tuning screws, apertures, iris, and posts).

There is huge flexibility of operation using the setup presented in Fig. 1. Batch or continuous flow reactors can be operated with little modification of the setup for a huge range of chemical reactions, in liquid, solid or gas phase. Depending on the process to be carried out, the user may need to procure a thermometer (optical fiber, IR or even a thermocouple for measuring the temperature at the outlet of the continuous flow reaction), a magnetic or mechanical stirrer, and a computer (for feedback, data logging and control).

Regarding stirrers, it has to be noted that at least the shaft of the mechanical stirrer must be made out of a material transparent to microwaves (such as Pyrex, PTFE, ceramic etc.); most PTFE stirrer shafts available in specialized laboratory catalogues are made out of metal coated with PTFE. These shafts will act as microwave antennas and conduct microwaves (leakage) outside the reactor. For magnetic stirring bars, their size must be chosen as such as they are always well immersed in the reaction mixture. The speed of stirring must be also optimized (strong vortexes may increase the reflected power).

2.4. Batch vs. conveyor applicators

Multimode cavities are made in at least two basic forms, namely oven type and conveyor belt, which are, by far, the most commonly used forms of microwave applicators. The microwave energy, carried by a waveguide or a transmission line into the cavity (see Fig. 1), is deposited into the lossy load placed within. The efficiency of such cavities is much lower than single-mode cavities, 30 – 80 %, and it depends on their design, geometry, and materials of construction. To minimize the non-uniformity of the energy distribution inside the cavity, a stirrer ('wave mixer') may be added. The most used mode stirrers are the turntable, carousel for circling samples, and fan with metallic blades. As mentioned above, equipment based on this design are available for use in the laboratory.

Despite the sophistication (e.g. monitored and regulated pressure and temperature), it is usually limited by the crude means of power control, i.e. switching the microwave power supply On and Off in a time frame of several seconds, and basically, no feedback on the microwave parameters. Up to present, all laboratory multimode microwave cavities, resonant or non-resonant, are fed by magnetrons and they are suitable for use with much larger volume samples than single mode cavities.

2.5. Safety measures

When using dedicated microwave laboratory equipment, either consumer or any other type, the operator must have handy a microwave leak detector, and must make sure that the level of leakage is below the one admissible by legislation (e.g. 5 mW/cm^2 at 5 cm from the source). Measurements must be done every time when the equipment is operated, as microwave leakages may appear if changing reaction conditions, type of reactor, equipment age, etc.

3. Basic approximations

When performing microwave-heating, the parameters to be taken in consideration are related to the sample as well as the equipment. The sample's parameters are related to its dimensions, dielectric properties and their temperature dependence, boiling point, viscosity, penetration depth, temperature distribution inside the sample, etc.. The equipment parameters are the power rating of the microwave components, the reactor selection suitable for the intended process, pulse or CW microwave generator, forward and reflected power, single or multimode applicator, temperature control, etc.

To enable proper operation of the equipment and reliability of the results, in addition to above considerations, simple 'finger rule' calculations and reflexions could be done before trying out new 'crazy ideas' using microwave equipment. For instance, the power P_a needed to heat up a sample (without phase transition) can be approximated by:

$$P_a = mC_p \, \Delta T / \Delta t \qquad (1)$$

where m is the sample's mass, C_p is the specific heat capacity, ΔT is the temperature increase, and Δt is the exposure period. For example, heating up 250 mL of ethanol ($\rho = 0.789$ g/ml, $C_p = 2.46$ J/g°C) from 15 to 61 ^{0}C in 2 minute, requires at least 186 W of microwave power.

Let us suppose the microwave equipment available for this test is rated at 800-W fixed power, the time to heat the sample would be shortened accordingly to 28 seconds. These basics calculations give a very fair idea of the minimal microwave heating parameters when starting a test; losses to the environment by heat transfer from the sample and the efficiency of the microwave applicator must be also taken into consideration.

It is easy to see from the above calculations that there is a certain flexibility in playing with these parameters to ensure reliable results. For example, if the rated power of the available equipment is high, choosing a higher volume of the sample to allow longer test time will lead to more accurate microwave power emission from the microwave generator.

Similarly, simple calorimetric measurements may help the operator to estimate the power absborbed by the sample. For example, placing a given amount of water in the same applicator in which the reaction will be done, and meauring the temperature increase (either in-site by optical fibers or ex-situ by a themorcouple, etc.), may provide an estimate, using Eq. (1), for the available microwave power.

An anlysis of published studies reveals a much higher power than rated by the manufacturer, which unfortunatelly is still the case of some commercial laboratory equipment[3].

4. Why not a domestic microwave oven?

Looking at published work, it seems that the microwave kitchen oven is the most used heating tool by chemists (I have not seen kettles or electric kitchen ovens used in chemistry laboratories). So why microwave consumer ovens? They are cheap and readily available in any supermarket, one can make holes in the metal walls, etc., but it also has significant drawbacks as follows:

(a) Running chemical reactions in consumer ovens is dangerous for the operator and for the equipment. The use of flammable or toxic solvents, which can easily reach boiling and leak inside the oven, metal catalysts, which can rapidly reach very high temperatures especially when working with small quantities are a few examples leading to fire inside the microwave cavity, explosions, burns, etc.

(b) Microwave heating pattern change with the oven model/manufacturer, and even with the position of the sample[4] (see Table 1).

(c) The oven is not suitable and becomes dangerous especially when heating small samples: due to the high power sent inside the cavity at oven ON (as shown in Fig. 2) small samples can overheat very rapidly, explode, melt the reactors they are placed in, especially those made out of PTFE, polyethylene, polypropylene (even if these are classified as 'microwave transparent materials).

(d) The microwave power delivered inside the oven is an average power and changes from one model to another.

(e) The magnetron can stop and restart without much warning to the user, the light is on, the turntable turns. A thermal snap switch mounted on magnetron's body turns off the magnetron if the temperature reaches 40 ^{0}C. Depending on the oven model the magnetron may auto restart once the temperature is lower than 40 ^{0}C.

(f) The standard construction features insure the magnetron with certain protection, e.g. most of the reflected power is dissipated within, if the oven is operated empty or with a small/non-absorbing load. The removal of the rotating plate, and holes making through the walls, result in microwave leakage, disturbances in the operation of the oven, which also change the heating pattern leading to random heating results and ultimately, to the destruction of the magnetron.

Table 1. Temperature increase variations for a 50-g Canola Oil sample in different microwave ovens

Oven Brand	Position on Turntable	Temperature (°C)		
		Initial	Final	Δ T (°C)
General Electric	Center	23	163	140
	Edge	23	71	48
Sharp	Center	24	76	52
	Edge	24	94	70
Kitchenaid	Center	26	129	103
	Edge	26	111	85

5. Conclusions

The number of laboratories around the world investigating the potential of using microwave heating to stimulate chemical reactions is increasing. Following up the research, seeing more and more contributions dedicated to chemistry at scientific manifestations like IMPI and AMPERE, but also as a chemist, I believe there is *a microwave effect* with high potential for the industry. I believe the microwave effect means all positive contributions to reaction rate enhancement, better process control, higher yield reaction products, and not at last, the excitement of a new technology, which can change the way the chemistry is done.

The industrial plant is not a laboratory – once production starts, it is difficult to stop and change one or two parameters to make it work. To assure that strategic goals are achieved, knowledgeable and experienced chemists, chemical engineers and microwave equipment designers must work closely with the end-users at the outset of the project, in order to clearly identify the project parameters. The greatest opportunity to impact project costs is at the beginning, during master planning and conceptual development. As the project progresses, this ability diminishes. The consequences of the decisions made in early phases of the project will dictate the viability of the project from a financial perspective, and also have long term effects on operations and cost of ownership.

The main drawbacks of the microwave-assisted chemistry are very often published to be related to the equipment cost and the low penetration depth of the microwaves (mainly at 2.45 GHz) in the reaction mixture. As for my part, I do not believe these are the real problems, and there are ways to overcome them, such as:

(a) Good mechanical stirrers, reactors in series or in parallel; continuous flow reactors; tailoring the microwave chemistry (choice of solvents, catalysts, energy input, etc.).

(b) Choice of a lower microwave frequency (higher penetration depth); higher power density (smaller reactors and potentially lower or no pressure); multiple microwave inlets; synergy with other heating methods; reactors made out of metal (rather than microwave transparent materials).

(c) Higher cost of investment (CAPEX) should not be looked at as simply as that. The economics are far more complex; in general, the end user will accept a higher CAPEX if the new technology can assure an overall improvement of at least 20% over conventional technology. In addition, many microwave assisted syntheses (especially organic syntheses), if carefully optimized, offer advantages like reduction in catalyst quantity, and reduction or elimination of solvents (which render such processes more environment-friendly and can justify higher CAPEX);

(d) For quicker success, the researcher must work closely with potential end users: they should address together ways to solve existent problems (e.g. treatment and disposal of chemical waste) or to develop new products, perhaps more in the area of specialty chemicals with high added value;

(e) Lastly, the integration of the microwave chemistry as a field of study in academic curricula may find now enough justification not only because it is a part of industrial reality but also because of its research and educational potential.

The introduction of 915 MHz and 2.45 GHz solid-state microwave generators, and all advantages they bring, ought to make an important impact in the way microwave-assisted chemical research is done. The possibility to control precisely the energy going into a reaction should help provide answers to still existing question marks and to promote the progress of microwave-assisted chemistry as a new or substituting technology for the chemical industry[5].

For further reading:

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4. R.F. Schiffmann, *Problems heating small samples in domestic microwave ovens*, 3rd Global Congress on Microwave Energy Applications (3GCMEA), Cartagena, Spain, July 25-29, 2016
5. Links to relevant websites:
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   www.impi.org
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Dr. Marilena Radoiu is a Chartered Chemist (CChem) and a Member of the Royal Society of Chemistry (MRSC). She received her M.Sc. in Organic Technological Chemistry from the Polytechnic University of Bucharest in 1993 and Ph.D. in Radiochemistry and Nuclear Chemistry from the same University in 1998.

Dr. Radoiu has extensive work experience in different international academic and industrial environments. She has worked for 20 years in various countries (Romania, Canada, UK and France) in the development of microwave-assisted technologies with applications to chemical synthesis, biomass extraction, plasma etc. Her work has included engineering and development of novel industrial and scientific standard and custom products, such as Zenith Etch and Sirius6000 (microwave plasma reactors for semiconductor gas cleaning), laboratory equipment such as MiniFlow 200 and Minilabotron 2000. Dr. Radoiu is also a member of several professional associations, including the Association for Microwave Power, Education and Research in Europe (AMPERE).

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