



State of the art of the fundamental aspects in the concept of microwave-assisted heating systems

Cristóbal Valverde^{*}, Margarita-Manuela Rodríguez-García, Esther Rojas, Rocío Bayón

CIEMAT-Plataforma Solar de Almería, Carretera de Senés, km 4, 04200, Tabernas, Almería, Spain

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ABSTRACT

As the processing of materials using clean energy becomes increasingly important, indirect conventional heating is being replaced by microwave-assisted heating. This method transforms energy directly into heat, providing rapid heating with instantaneous start-up, improved control and homogeneity of temperature distribution, and enabling a compact design. Therefore, the impact of microwaves in industry is growing in recent years. The complexity of incorporating microwaves into any industrial process resides in the need for a customised design. This is emphasised in the case of continuous flow microwave heating systems where the electromagnetic distribution and its transformation into heat, as well as the dynamics of the fluid to be heated under different operating conditions, are solved. For this purpose, and apart from the dielectric and magnetic properties of the material to be heated, which define how it behaves in relation to electric and magnetic fields when exposed to microwaves, there are several factors influencing the design of the process: the microwave generator and the transmission line, the geometry of the applicator where the electromagnetic field is distributed, the material container of the sample or the temperature measurement techniques to control the process. Additionally, numerical simulation is an essential tool for designing and predicting the suitability of any microwave system. This is the case of composite materials processing, where the application of numerical simulation tools arises from the need to achieve advanced materials more efficiently. In this paper, these fundamental aspects for the design of a microwave-assisted heating system are reviewed, presenting examples of various applications where this microwave technology is integrated.

1. Introduction

Microwaves are electromagnetic waves in the frequency band between 300 GHz and 300 MHz, i.e., with wavelengths in the range of 1 to 1000 mm [1]. Although they do not constitute ionising radiation, these waves can interact directly with the matter at the molecular level, varying its electric and magnetic fields, resulting in the heating of the sample [2].

In the last decades, the use of microwaves for heating applications has exploded as resource efficiency and sustainable processing become more important, as it allows the use of renewable energy throughout the process. Noteworthy is the use of solar and wind energy to power microwaves in chemical reactors [3]. This is an increasingly cheaper alternative to conventional fossil-fuelled chemical reactor heating [4]. The main advantages of this method, as opposed to conventional heating, are that the energy is converted directly into heat inside the sample, rather than transferring that heat to the sample [5–10]. Moreover,

heating rates of up to 400 K/min can be achieved [11], as well as no direct contact between the heat source and the material to be heated, greater control with instantaneous stops and starts, and reduction of equipment size and heat waste [12,13].

Several frequencies – 13.56 MHz $\pm$ 6.68 kHz, 27.12 MHz $\pm$ 16.0 kHz, 40.68 MHz $\pm$ 20.0 kHz, 433.92 MHz $\pm$ 87 MHz, 915 MHz $\pm$ 25 MHz, 2450 MHz $\pm$ 50 MHz, 5800 MHz $\pm$ 75 MHz, 24,125 MHz $\pm$ 125 MHz – are set for industrial, scientific and medical (ISM) use [14], however, it is 2450 MHz frequency magnetrons that are used for most applications. These magnetrons provide more efficient electromagnetic energy-to-heat conversions due to the higher performance factor of up to 86% efficiency in modern designs. Magnetrons with 915 MHz compared to the 2.45 GHz frequency can heat larger samples as they offer a higher depth of penetration (D_p) [15,16]. Marinell, et al. [17] developed a 915 MHz single mode cavity system for sintering large samples of low-loss dielectric alumina. The process is based on controlled hybrid heating with two SiC susceptor plates, placed parallel to the electric field to

^{*} Corresponding author.

E-mail address: cvalverde@psa.es (C. Valverde).

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avoid plasma and thermal instabilities, and within a thermal insulation material. Following this idea, Rosa, et al. [18] highlighted some research opportunities in the field of carbon combustion synthesis, specifically, synthesising nanostructured composites, exploring less conventional frequencies, e.g. 5800 MHz, corresponding to higher and lower penetration depths. At the same time, new ways of producing microwaves are emerging, using solid-state (semiconductor) technology, which offer controllable radiation to the product load, achieving more uniform heating compared to magnetrons [19,20].

1.1. Brief state of the art of microwave-assisted heating applications

This technology offers great possibilities in different sectors [21]. More and more applications for microwave heating are emerging as more and more research is done on how microwaves interact with different materials, their physical mechanisms, and how they can be used for heating [22,23]. This method depends on the dielectric properties of the material to be processed, which measure the response of the material to an electric field, while its magnetic properties affect the capacity of the material to store and convert the energy of the magnetic field into heat [24]. Concerning the sample to be heated, the mass/volume to be treated, whether the sample is heterogeneous, initial, intermediate and end products, as well as its shape and location in the cavity, also have an influence [25]. In terms of equipment, the variables to have an impact on heating are the geometry, its dimensions, the microwave source and the energy transfer system, the selection of the material for the cavity and also the holder of the sample treated [19,25–29]. Microwave technology is used in different applications, which can be classified according to the required temperatures [13]. Table 1 presents a classification of microwave heating applications into three main groups according to temperature, based on the work of Mishra et al. [13].

To understand the impact of microwave heating on industry, the number of publications has been quantified using the Web of Science (WOS) online database. Fig. 1 shows the number of scientific publications on industrial microwave heating, their time evolution over the last 15 years, and the total number compared to other technologies. These are obtained by keywords in the WOS core collection. These keywords are “industrial process”, “heating”, “microwaves”, “flow” and “numerical simulation”. It can be seen that the number of articles has increased considerably over the last 7 years, reaching a record high of 85 articles in 2021 compared to 16 articles in 2009. The total amount represents almost 10% of all articles researching heating processes in industry. Among the total number of articles based on microwave heating processes, only 2% are articles where numerical simulation is the basis of the research conducted. In addition, microwave heating processes can be performed for static materials inside a cavity or in movement, i.e. a liquid or gas flowing through a carrier tube. In the last 15 years, the

number of papers on heating of moving fluids with microwaves represents 8% of the total number of papers on industrial heating.

1.1.1. Examples of continuous flow microwave heating systems for different industries

Continuous flow microwave heating is a very promising method for industries such as food and chemical engineering. It is a multiphysics approach, in which, unlike static microwave heating, not only electromagnetic distribution and its transformation into heat is involved, but also the fluid dynamics. To improve process efficiency, uniformity and adaptability under different operating conditions, there are new variables to investigate, such as flow rate, carrier tube geometry, number of tubes, use of spiral tubes, their location and orientation. Despite the many advantages of microwave heating, it is difficult to find scientific publications related to systems operating at moderate and high temperature, especially if the material to be heated is a moving fluid. That is not the case at lower temperatures. There are several works for the food industry. Topcam et al. [49] investigated the optimization of the cavity geometry and process design on an industrial scale in continuous flow processes for temperatures up to 60 °C. The aim is to avoid inhomogeneity in heating due to the limited D_p in the medium by microwaves. The systems used were cylindrical single mode cavity with only one magnetron of 915 MHz frequency, and multimode rectangular cavity with 8 magnetrons of 2450 MHz placed on the walls, close to the pipe. With another orientation in the second system, the results showed non-uniformities due to local temperature increases. The effect of the number of tubes inside the cavity and their orientation, the applied power and the fluid velocity were studied by multiphysics simulation using COMSOL Multiphysics software, the liquid sample being a whole liquid egg. It was shown that the elliptical cavity results in a more uniform temperature distribution. At the same time, it was shown that larger cavity dimensions for processing a larger sample volume, as we will see in section 2.1.2, are valid for continuous flows. In the same area of liquid heating for the food industry, Tuta et al. [50] worked with a unique single mode cavity and helical tube design, which tested different fluids at different flow rates by numerical simulation and subsequent experimental validation. Zhu et al. [22] also worked with numerical simulation to understand the effects of dielectric properties, in this case working with food liquids (apple sauce, skim milk, and tomato sauce) by varying different parameters such as location or dimension of the applicator inside the cavity with different diameters. More specifically, microwaves are proposed as an alternative to conventional heating for the aseptic processing of low-acid vegetable purees. Kumar et al. [51] studied in the scale-up of a pilot prototype to industrial scale, with extended run times of 8 h, avoiding process-related problems such as inhomogeneity in temperature distribution. The process scale-up of green pea puree and carrot puree resulted a success in a 60 kW microwave system and static mixers installed at the exit to homogenise the temperature distribution. The following figure, Fig. 2, shows some of these unique low-temperature continuous flow microwave designs for the food industry.

There are also publications on continuous processes at low temperatures in the synthesis of chemical compounds. Baker-Fales et al. [39] focus on a microwave-assisted continuous flow-scale reactor for liquid-phase chemical processing. In this work, a microwave-assisted scale reactor for the production of 5-hydroxymethylfurfural (HMF) from fructose dehydration was studied, with specific application to the production of 5-hydroxymethylfurfural (HMF). The microwave source was a variable frequency solid-state generator (900 W, 2.45 GHz as reference frequency) connected to a configurable Malachite H-field cavity via a WR340 waveguide. The device included a 3-stub tuner and a sliding short circuit. This scaled-up reactor demonstrated an HMF yield of around 55% and 8 times the productivity of a conventional system, while maintaining high energy efficiency and a CO2 emission reduction of >60%. In addition, the experiments were validated using a computational fluid dynamics (CFD) model. Other uses where scaling of

Table 1
Classification of microwave materials processing according to temperature range and application [13].

Range of temperatures	Relevant applications	Refs.
Low Temperature Processing ($T < 500\text{ }^{\circ}\text{C}$)	Polymer, Polymer-matrix Composites (PMCs), Rubber	[30–38]
	Chemical, Medical	[39–48]
	Cooking, Drying	[22,49–60]
Moderate Temperature Processing ($500\text{ }^{\circ}\text{C} < T < 1000\text{ }^{\circ}\text{C}$)	Ceramics, Glass	[11,61–63]
	Low melting point metal-based materials	[64–66]
	Synthesis of Carbon Nano Tubes (CNTs) or other nanocomposites	[18,67–69]
High Temperature Processing ($T > 1000\text{ }^{\circ}\text{C}$)	High density ceramics, Ceramic-matrix Composites (CMCs)	[17,70–73]
	High melting point metallic materials, Metallic-matrix Composites (MMCs)	[74–79]
	Nano materials	[72,73,80–84]

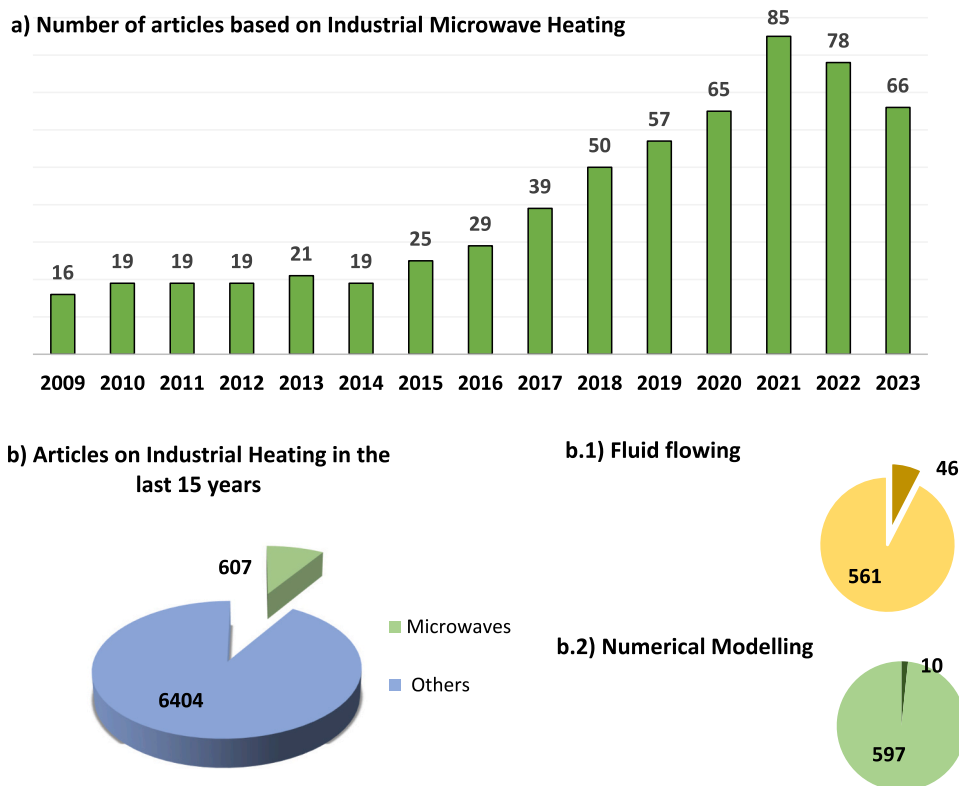


Fig. 1. (a) Time trend in the number of publications on microwave-based industrial heating, (b) total number of these publications out of the number of publications on industrial heating in the last 15 years, (b.1) articles whose research is on numerical simulations, (b.2) articles dealing with microwave heating of moving fluids.

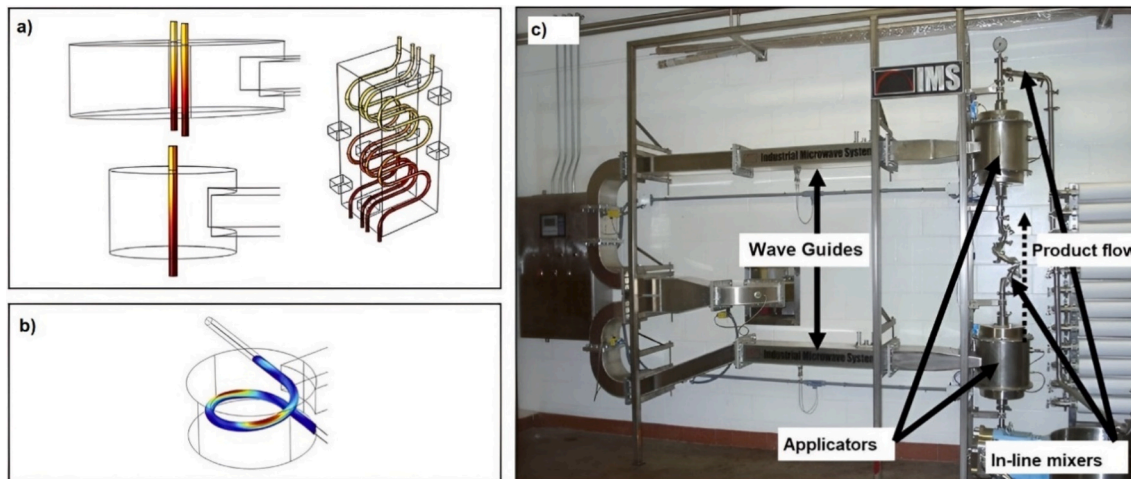


Fig. 2. (a) Cylindrical and elliptical cavity simulations with a single 915 MHz magnetron, and rectangular cavity with 8 magnetrons and tube with wave geometry [49], (b) unique single mode cavity and helical tube design [50], and (c) 60 kW microwave system for the aseptic processing of low-acid vegetable purees [51].

microwave systems is addressed include organic [40] or zeolite synthesis [41]. Research was also conducted on the use of the microwave cavity itself as a reactor, whose metal walls were adapted to continuous chemical reactions at high pressures of up to 7 MPa at temperatures of 200 °C [42]. The design was optimised and simulated with multiphysics simulation software by coupling wave propagation to heat transfer, and wave reflections were minimised, with the microwave energy being entirely absorbed by the reaction medium. In the same microwave reactor line, Saggadi et al. [43] developed a microwave device for chemical reactions with a single mode applicator that allows a uniform electromagnetic field. This setup resulted valid for the continuous chemical synthesis of quinoline from glycerol at temperatures up to

200 °C and high pressures up to 19 bar, with an important advantage being the long residence times or relatively high mass flow rates over 1 kg/h due to its large reactor volume. Comparing this pilot microwave reactor with conventional heating, the reaction time and energy consumption are shorter (32 min and 11,014 kJ/mol, compared to 112 min and 452,200 kJ/mol), and the quinoline yield is about 41% and 37% in the case of conventional heating. The following figure, Fig. 3, shows examples of these microwave reactors.

Among the most relevant recent high-temperature microwave publications are those addressing the pyrolysis process. Chao et al. [85] analysed microwave-assisted heating in the preparation of cerium oxide micro-nano particles, obtaining a better finish than conventional

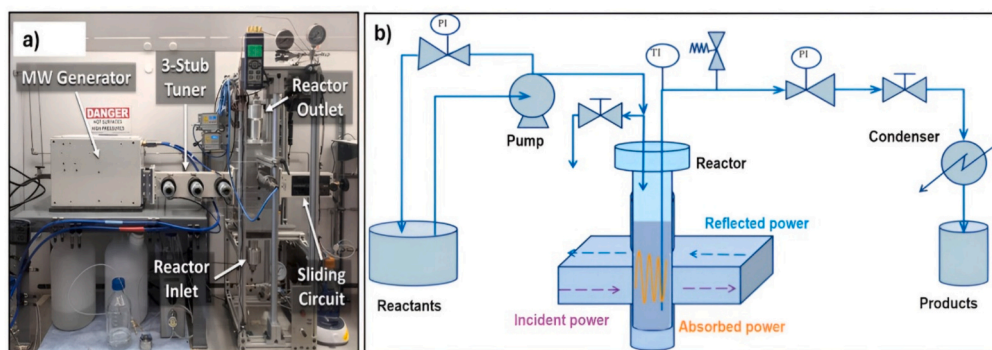


Fig. 3. (a) Microwave-assisted reactor with specific application to HMF production [39], and (b) diagram of continuous mode microwave pilot reactor for synthesis of quinoline from glycerol [43].

methods, in an efficient way. These conventional methods are based on contact heat transfer in fixed bed reactors, fluidised bed reactors or electric furnaces [86]. This microwave system used a Venturi reactor inside the microwave oven where microwaves were radiated by three equidistantly distributed magnetrons connected to this cavity with three BJ26 rectangular waveguides. In addition, numerical simulation was used to couple the various physics involved and determine the effects of microwave power, reactor location and waveguide arrangement. These factors modified the heating pattern, and it was observed that the degree of crystallinity and purity of cerium oxide improved with increasing microwave power. The following figure, Fig. 4, represents the diagram and model of the equipment used.

Anis et al. [87] also studied the application of microwave-assisted pyrolysis in the production of liquid biofuel from oil. In this work, a 900 W microwave reactor with 300 g of commercial particulate charcoal was fed with used cooking oil or fresh cooking oil at various feed rates ranging from 0.051 kg/h to 0.306 kg/h and temperatures 450 °C – 550 °C. The reactor used nitrogen gas to remove oxygen within the reactor, ensuring the pyrolysis condition. Microwave irradiation for pyrolysis and distillation processes proved to be effective in the conversion of used cooking oil into liquid biofuels, especially green diesel. Microwaves and radiofrequency (RF) radiation have been widely studied and applied for the treatment of raw oil [88]. Rosin et al. [89] worked on decreasing the viscosity properties of oil by means of microwaves in a thermal process and reduction of energy consumption. The reactor is a laboratory-scale continuous cavity designed with a microwave window, simulated and validated in experimental tests, to operate at pressures up to 3 MPa, ensuring several key functions such as flow conditions, safety and energy transfer. In the synthesis of organic compounds, polymers, inorganic materials and nanomaterials, microwaves stand out from the conventional method with higher performance and precision in the preparation of desired catalytic and organic materials, controlling parameters such as temperature, pressure and temperature gradient [30]. In the same field, Morschhäuser et al. [90] described a continuous flow microwave system based on a short-

circuited waveguide as a reactor capable of operating at 310 °C and 6 MPa, using corundum as a transparent container material. This reactor was able to process chemical transformations with power outputs of 0.6–6 kW and flow rates of 3.5–6 l/h. There are more microwave designs in chemical reactors operating at high temperatures for chemical synthesis [44,45], waste gas treatment [46] or hydrothermal liquefaction of biomass [47], and others. Many of these processes require the presence of a solid catalyst within the reactor, which is exposed directly to microwaves in an efficient process that reduces the operating temperature in some cases to 1200 °C as waste gas treatment, since reaching the combustion temperature of the catalyst intensifies the process. The following figure, Fig. 5, presents two of the microwave applicators applied for the treatment of raw oil.

1.1.2. Comparison of microwave heating over conventional heating in specific applications

As discussed in the examples of the different applications, most of them have advantages over conventional heating. The main advantages common to most cases are time and energy savings, as well as uniformity in temperature distribution which can lead to products with better properties. The table below, Table 2, presents the comparison of microwave-assisted and conventional heating in terms of time and energy consumption, as well as other advantages in some application examples.

The aim of this review article is to present how the fundamental aspects to consider in the design of a microwave system have been applied to specific industrial applications, highlighting the technological solutions implemented. This paper is organized as follows: within section 2 where the fundamental aspects of microwave system design are analysed, the first subsection (2.1) deals with the main hardware of microwave systems: microwave generators (2.1.1), transmission lines (2.1.2) and cavities (2.1.3). The latter is divided into two further subdivisions: single mode cavities (2.1.3.1) and multimode cavities (2.1.3.2). The second (2.2) explains how matter interacts with microwaves, the mechanisms involved in heating, and their classification. A short section (2.2.1) is dedicated to aspects to be considered in microwave-transparent materials to contain the samples. The third subsection (2.3) presents the techniques used for microwave heating, from direct radiation on the material to hybrid or selective heating for more specific applications. The subsection 2.4 describes the technology used in microwave systems for temperature measurement. Numerical simulation tools are presented in section 2.5, and finally the section 3 and section 4 summarises the main conclusions and introduces new research opportunities. While reading the paper, some concepts appear that are analysed in detail in the following sections.

2. Fundamental aspects of microwave system design

In order for microwaves to be absorbed by matter and transformed

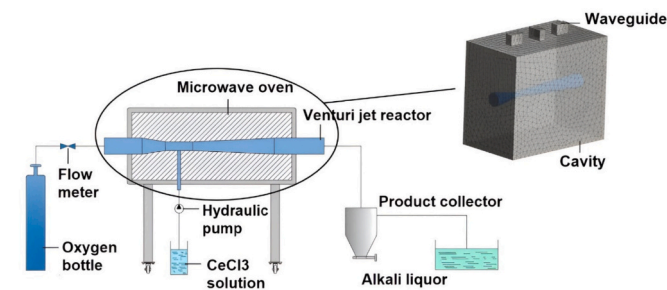


Fig. 4. Diagram and model of the microwave system developed to prepare cerium oxide micro-nano particles [85].

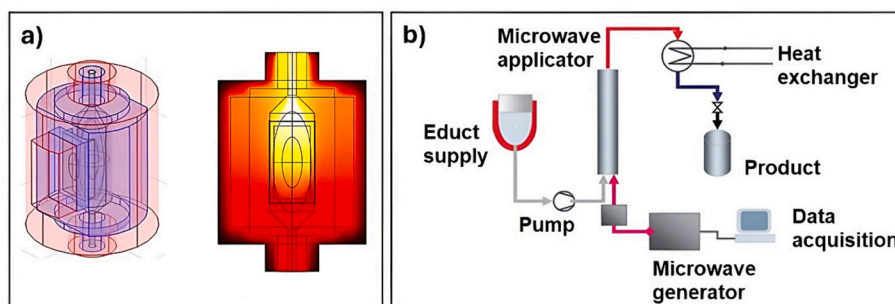


Fig. 5. (a) Simulation of the laboratory-scale reactor used by Rosin et al. [89], and (b) scheme of the continuous microwave synthesis designed by Morschhäuser et al. [90].

into heat, several factors influence the process and therefore ‘tailor-made’ design is required for each industrial application. Dabrowska et al. [84] described different self-designed microwave reactors for the synthesis of nanomaterials and other applications at laboratory and industrial scale. In the same line, Mishra et al. [13] identified the keys in the design of microwave-assisted systems for processing advanced materials such as the design of a suitable susceptor around the target material for a higher diffusion rate or a special tooling for the control of the microwave process. The following diagram, Fig. 6, summarises the main points to be addressed in the design of a microwave system, common to all applications, and which are discussed in detail with specific examples in the following sub-sections.

2.1. Microwave generators, transmission lines and applicators

Although each process requires its own design for effective operation, they all share the same main components: a microwave generator, the power transmission and the applicator/cavity containing the sample to be treated [25].

2.1.1. Microwave power generation

As mentioned in Section 1, the sources of microwave for industrial applications are magnetrons and solid-state generators. In the case of magnetrons, their maximum power rating and electrical efficiency depend on the geometry of the magnetron and the quality and manufacturing precision of the components. Assuming good magnetron quality, an electrical efficiency of 70% can be achieved for 2.45 GHz magnetrons, while up to 90% can be reached for 915 MHz magnetrons. The lifetime of a magnetron expressed in total number of microwave hours is >8000 h [25]. Solid-state technology provides precise frequency control [4] and output power up to 300 W at 2.45 GHz and 700 W at 915 MHz [3]. However, compared to magnetrons, for the same power level, the cost is more than two to four times higher due to the complexity of the RF circuitry and materials, mainly the sophisticated low loss printed circuit board (PCB) substrates. In contrast, solid-state generators can operate uninterruptedly for 15 years without performance degradation [5]. Zhou, et al. [60] provided fundamental guidance on designing and modelling microwave heating systems based on solid-state generators. They used a domestic microwave oven to compare the frequency spectra (i.e. peak frequency and bandwidth) of the magnetron and the solid-state generator. While magnetron microwave spectra vary depending on the sample to be heated and its position, the ability of solid-state generators to control the operating frequency precisely makes this source of microwaves a key element in the development of the next generation of microwaves. The methodology for measuring the spectral quality of the solid-state generator relied on a spectrum analyser to measure microwave leakage from the oven. The antenna measured signals five times per second at two positions, 5 cm away from the microwave oven, to assess the influence of position. For three representative food loads and a maximum of 300 snapshots of

frequency spectra during 60 s of microwave heating, it was shown that the measured peak frequencies exactly matched the set frequencies, independent of the food loads and their positions. In the same work, a simulation model was developed with COMSOL software that supports the results obtained by the solid-state generator with more stable and predictable heating patterns.

2.1.2. Microwave transmission lines

In a microwave system, the radiated energy travels from the source to the cavity through a waveguide or coaxial cable, which are the most common transmission lines. A waveguide is a rectangular, circular or rigged hollow structure made of metal such as copper, aluminium or other metal with high thermal and electrical conductivity. The rectangular type is the most common as it supports the propagation of transverse electric (TE) and transverse magnetic (TM) modes. Its dimensions are standardised. At 2.45 GHz the waveguides used are WR284 (72.130 mm × 36.065 mm), WR340 (86.360 mm × 43.180 mm) and WR430 (247.500 mm × 123.750 mm), and at 915 MHz WR975 (247.500 mm × 123.750 mm) [25]. Ellison et al. [48] developed a microwave pyrolysis system for the thermochemical conversion of biomass into chemicals and biofuels (bio-oil and syngas), where the waveguide was used as a single mode cavity. This system implemented continuous phase shifting by means of a sliding short circuit to mitigate standing wave effects and achieve better heating uniformity. The figure below, Fig. 7, shows the experimental set up.

Coaxial cable, consisting of a cylindrical conductor commonly made of copper, an air-like dielectric and a conductive screen, is capable of propagating a transverse electromagnetic (TEM) wave. The maximum power supported increases with size and the electromagnetic waves propagate at a frequency always below the maximum cut-off frequency. The power losses along the cable depend on the diameter and length required to connect the source to the cavity and are therefore important design parameters to consider in the TEM power absorbed by the sample. In addition to the transmission line itself, it is important to design the microwave transport system to ensure that the sample absorbs all the microwave power from the generator.

Any design must control the reflected power, as high levels of reflected power can create arcing and damage the generator. A waveguide or coaxial circulator is a three-port device intended to create isolation between transmitted and received signals. To absorb the reflected power, a separate “dummy” load is connected to the circulator. Ideally, this circulator should not attenuate the power.

2.1.3. Microwave applicators/cavities

Microwave applicators are structures, mostly metallic, that confine the microwave energy within the space where the sample to be processed is placed. The distribution of these microwaves inside the chamber are governed by Maxwell’s equations [98].

$$\nabla \times E = j\omega H \quad (1)$$

Table 2

Comparison of time and energy savings and other significant advantages of microwave heating over conventional heating in some applications.

Examples of applications	MW vs Conventional heating			Refs.
	Time consuming	Energy consumption	Other aspects	
Chemical reactions with microwave (continuous chemical synthesis of quinoline from glycerol)	32 min compared to 112 min	11.014 kJ/mol compared to 452.200 kJ/mol	Quinoline yield is about 41% with microwaves and 37% in the case of conventional heating	[43]
Microwave treatment of raw oil	N/A	N/A	Viscosity reduction by the conventional heating method is reversible, requiring heating equipment installed along the pipeline at a higher cost.	[91]
Microwave synthesis of micro-nano particles	Up to 10 s compared to about 2 h for the conventional method	N/A	In the case of ZrO ₂ particles, better dispersity of the particles via microwave heating whose size was < 40 nm compared to conventional heating that exceeded 60 nm	[92]
Microwave melting of metals Tin Lead Aluminium	MW - Conventional 5 min - 11 min 6 min - 14 min 9 min - 29 min	2 to 6 kW compared with induction heating which required 10 to 150 kW		[74]
Hybrid heating with microwaves (powder compacts of metal-based materials)	0.08–0.33 times compared to conventional sintering	N/A	Improved tensile properties due to less exposure to high temperatures.	[93]
Microwave sintering with ceramics	10 min compared to 1 h	N/A	Density of 99.9% with microwaves compared to 98.0% with conventional sintering. Also superior Vickers hardness (16.0 GPa) compared to conventional sintering (13.4 GPa)	[94]
Drying of ceramics (silica sludge)	15 min compared to 450 min	N/A		[95]
Microwave curing of carbon fibre reinforced polymer (CFRP) composite through resonance structures	156 s or 30 s (microwave power level of 6 or 12 kW) compared to 1120 s	Reduction of 99.2%, i.e., 0.26 kWh or 0.1 kWh to 18.4 kWh with the autoclave	The flexural strength and the interlaminar shear strength (ILSS) resulted slightly higher, 4.28% and 6.33% respectively	[37]
PMCs with microwave (carbon fibre epoxy composite)	50% cure cycle time reduction	N/A	Shear strength 9% higher Lower void content (< 2%)	[96]
Hydrogen production with microwaves (catalytic dry reforming of methane)	N/A	4.6 kWh/m ³ of H ₂ with MW compared with 9.47 kWh/mol conventionally	11% (over 10Ni/AC) and 8% (over Ni/MgO/AC) higher efficiency	[97]

$$\nabla \times E = -j\omega\epsilon_0\epsilon^*E \quad (2)$$

$$\nabla \cdot (\epsilon E) = 0 \quad (3)$$

$$\nabla \cdot H = 0 \quad (4)$$

where E is the time-harmonic electric field (V m⁻¹), H is the magnetic field (A m⁻¹), ϵ^* is the complex permittivity of the material (F m⁻¹), ϵ is permittivity, ϵ_0 is permittivity in air, μ is magnetic permeability of microwaves (H m⁻¹) and ω is the angular frequency.

By calculating the electromagnetic field pattern, with the system's boundary conditions, the sample can be positioned in an area of maximum electromagnetic field strength, ensuring rapid processing [26,99,100]. These microwave applicators are classified into two groups according to their size: single mode or multimode cavities. The following figure, Fig. 8, shows how the electric field is distributed in both types of cavities.

2.1.3.1. Single mode microwave applicators. These applicators are closed structures of size comparable to the microwave wavelength in which only one electromagnetic mode is excited. Its design is based on Maxwell's equations so that the wave mode resonates at the excitation frequency. In this case, the distribution of the electromagnetic field can be accurately predicted, allowing the sample to be placed where the field is at its maximum strength for faster and more homogeneous heating. However, its dimensions have the disadvantage of a smaller volume of material that can be heated. The amount of material can be increased with cavities designed to operate at lower frequencies (e.g. 915 MHz instead of 2450 MHz) [101]. Furthermore, the dielectric properties of the material, which are responsible for the absorption of electromagnetic energy, vary as the material heats up. This influences the resonant frequency and therefore requires a tuning system to adjust or tune the resonant cavity. This is the case for Lewis et al. [102] who implemented an E - H tuner to a single mode cavity. With stepper motors, this control system has the ability to continuously change the position of plungers within the waveguide to obtain a lower inverse power reading. These

plungers function as variable short circuits and therefore, it is possible to change the cavity dimension. Oliveira and Silva [103] developed a waveguide section delimited by a short-circuit plunger and an iris, inside which a thin ceramic cylinder is inserted. This model, with iris opening and short-circuit position, provided accurate results, thus solving the thermoelectromagnetic problem of heating ceramic samples.

Among the applications where this type of microwave applicator is used include the processing of phase change materials (PCMs). This physical phenomena has been investigated numerically by developing models in one dimension [104], and two dimensions [105] in a cylindrical cavity. Ratanadecho et al. [52,53] makes use of a rectangular waveguide powered by a 1000 W magnetron to predict the temperature reached and melting front in multilayer packed beds of glass beads and water or ice, by applying a single transversal electric (TE₁₀) mode and frequency of 2.45 GHz. The developed model, its results for different layer positions, agreed with the experimental results. This rectangular waveguide microwave system was also used for the drying of multilayer porous materials. [54]. Depending on the pore size and moisture content of porous materials, their electromagnetic distribution within the waveguide varies, which affects the wave penetration and thus their absorbed power. The developed model predicts the microwave behaviour observed in the experiments, which improves the understanding of the microwave drying process. In this specific case, the smaller the pore size, the faster the drying speed due to higher capillary pressure. In cases such as the heating of granular materials with low thermal conductivity, microwaves are advantageous over any other method. These cases may be necessary to evaporate liquid contents. Examples are the remediation of organic contaminants from soils or rock fragments, and the extraction of organics from oil sands, oil shales and biomass. When the target compound is organic, the water contained in the sample acts as a receiver, allowing heat transfer to the organic contaminants. Robinson et al. [106,107], using single mode cavity, demonstrated the great potential of microwaves in the case of oil recovery from contaminated drill cuttings. Harutyunyan et al. [69] worked on the purification of single-walled CNTs, making use of tuned single mode cavity microwaves inducing the TE₁₀₃ wave mode with power of 1.5 kW and 2.45 GHz. This

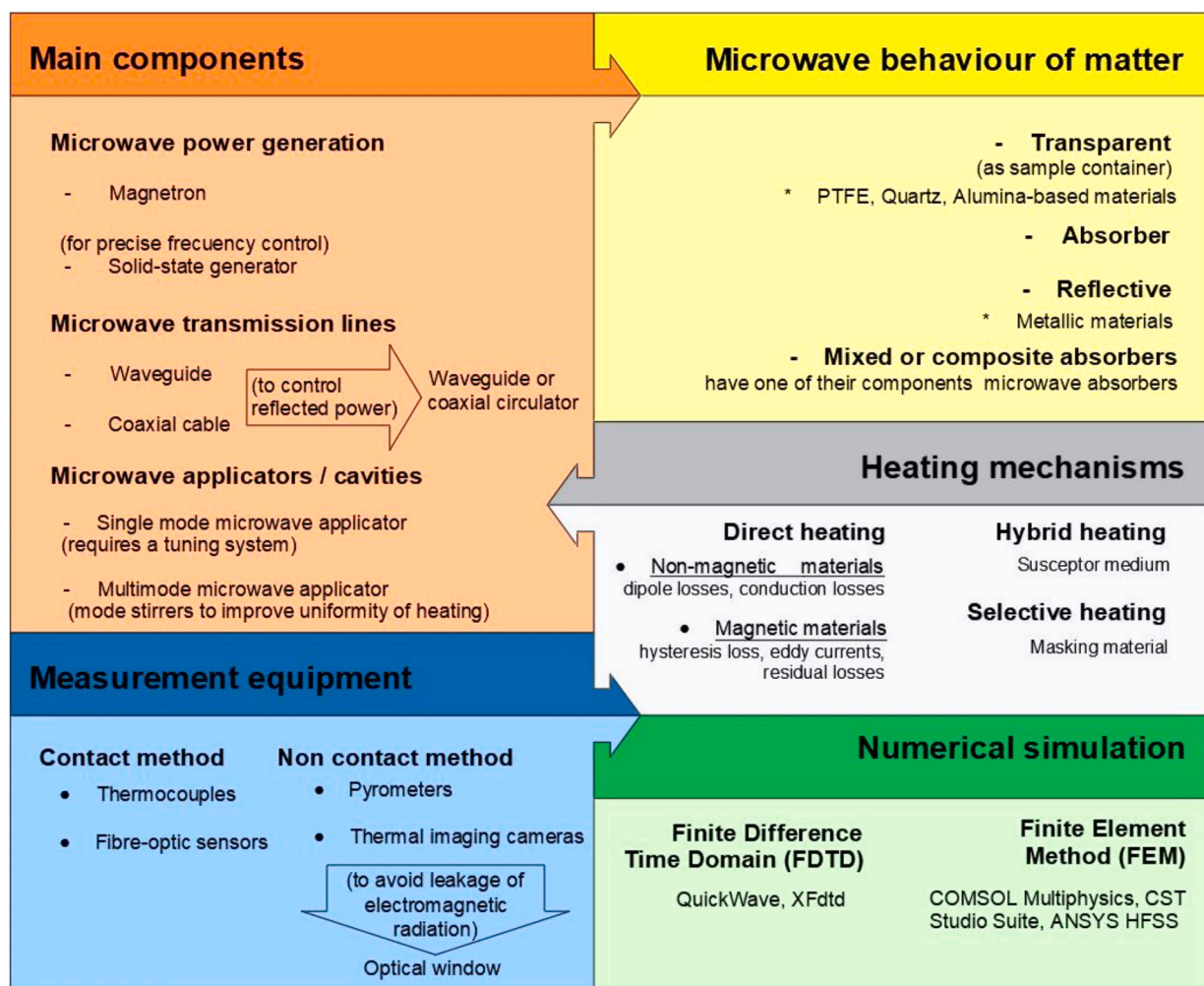


Fig. 6. Diagram of the main issues to be addressed in the design of a microwave system.

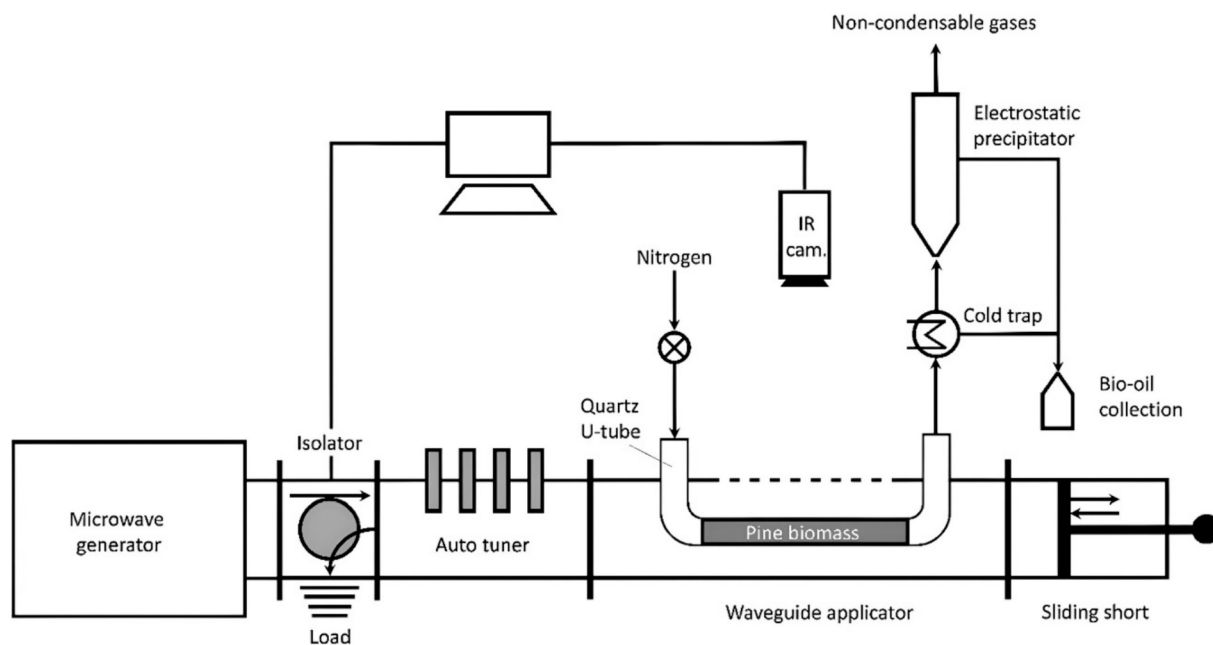


Fig. 7. Experimental set up of a microwave pyrolysis system based on a single mode waveguide [48].

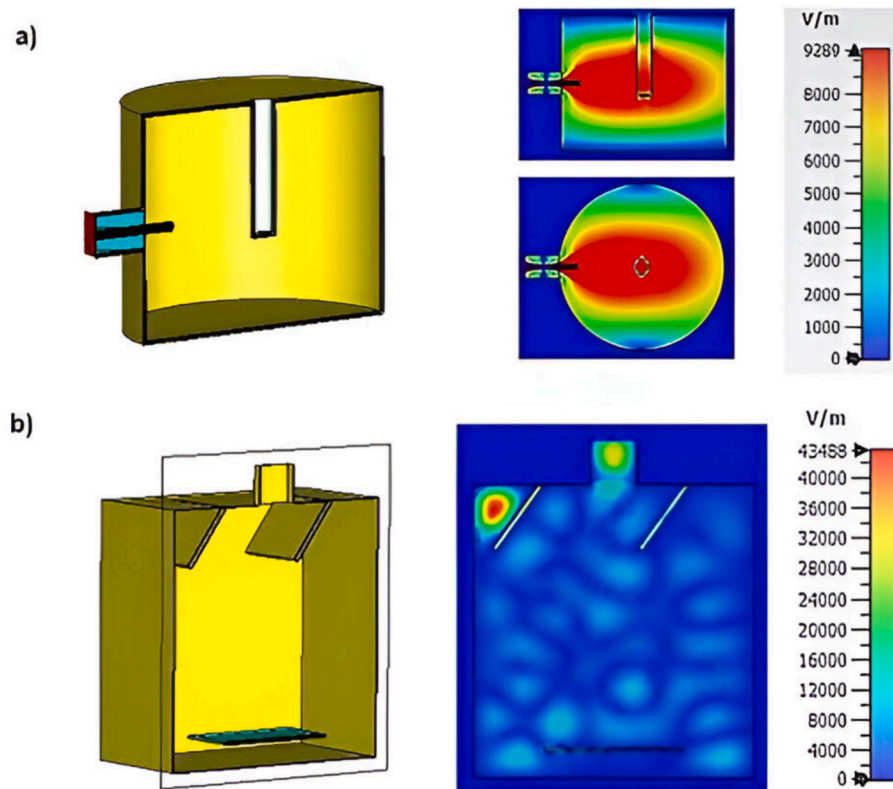


Fig. 8. Examples of a) single mode applicator with coaxial probe. Plan and profile view of the electric field distribution; b) multimode applicator fed with a magnetron and with mode stirrers or stirrers. Section view of the electric field distribution (software: CST Studio Suite).

local heating (up to 500 °C) weakens or removes the protective carbon layer, allowing acid treatment and removal of metal catalyst residues. Microwaves with such cavities have been successfully applied in other applications such as sintering of metallic and ceramic materials [61], or continuous flow processing applications [108,109]. Catalá-Civera et al. [110] designed a microwave-based system for the measurement of dielectric properties of materials up to temperatures of approximately 1000 °C. This system uses a cylindrical cavity with two electromagnetic sources and a cross-coupled filter to isolate the two wave modes, TE_{111} with resonant frequency (~ 2.432 GHz) for sample heating, and transverse mode (TM_{010}) at the same frequency for measurement. The following figure, Fig. 9, shows a schematic of the equipment designed [111]. The sample permittivity is calculated using an improved cavity perturbation method (CPM) based on the change of the resonance in the cavity when a material is introduced. There are other techniques to measure the permittivity of materials classified as resonant or non-resonant [112], as well as resonant cavities [113], open resonators [114], and dielectric resonators [115]. The improved CPM method ensures an overall accuracy of $\pm 1.3\%$. In turn, a proportional – integral – derivative (PID) control algorithm adjusts the heating rate by adjusting the bandwidth of the sources, in this case, with solid-state technology. Pérez-Campos et al. [116] used this dynamic permittivity measurement with dual-mode cylindrical cavity and frequency near to 2.45 GHz to characterize ground tire rubber (GTR). The results showed the high dependence of temperature, heating rate (power) and energy delivered with the GTR dielectric properties. Thus the samples experienced abrupt changes in their dielectric parameters at 160 °C and 190 °C, and after cooling the samples the permittivity values were different due to devulcanization.

2.1.3.2. Multimode microwave applicators. These are applicators with a size larger than the microwave wavelength, with multiple zones of electric field strength maxima and minima, where the multiple wave

modes present within the cavity interfere. Their design, unlike the previous ones, is often based on experience [26]. In this type of applicator, hot spots form in the peak areas. By increasing their dimensions, this number of hot spots increases, which affects the uniformity of heating. To avoid this undesired effect, the most widely used solution is the installation of mode stirrers inside the cavity [117,118]. In order to avoid large microwave leakage, Monzo-Cabrera, et al. [119] designed a high-power coaxial filter for microwave ovens. This filter, with two-port coaxial cavities coupled via coaxial lines, allowed the introduction of metallic stirrers with attenuations up to 70 dB obtained by numerical simulation and experimentally.

This type of applicator has been used for various industrial applications. For example, there are many works available in metallurgy with microwaves in multimode cavities. Of interest are those aimed at improving the mechanical properties of magnesium from synthesis with copper nanoparticles (NPs) [80], alumina [81], silicon carbide [82], or yttrium oxide [83] as a reinforcement. Although magnesium is an abundant element on Earth, very light, with high hardness, easily machinable and has a lower cost, its use in industry is still limited compared to aluminium, because of its low elasticity and resistance to high temperatures, as well as being highly corrosive due to its low electrical potential. The melting temperature of magnesium is 650 °C. In all these cases, rapid microwave sintering was advantageous as a step prior to hot extrusion to synthesise near-dense magnesium compounds. The results revealed an improvement in their hardness, yield strength, ultimate fracture toughness, or increased ductility as the amount of NP reinforcement increased. Chandrasekaran et al. [74] studied the melting of lead, tin, aluminium and copper using silicon carbide as an absorber in a hybrid heating process. Different power levels from 520 W (40%) to 1300 W (100%) were used for an operating frequency of 2.45 GHz, and temperature range up to 1083 °C. Compared to conventional 2500 W muffle furnace melting, microwaves require less time and energy. Microwaves have also been used for the synthesis of CMCs. The interest in

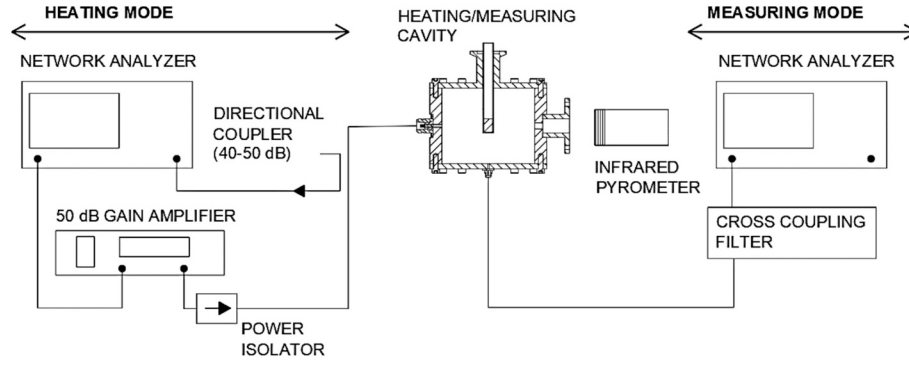


Fig. 9. Scheme of testing device for dielectric properties, using a cylinder cavity and dual mode [111].

ceramic nanocomposites is due to the advantages they offer in high-temperature applications such as high hardness, high elastic modulus, strength and ductility, as well as low chemical wear, among others. Multimode cavity microwaves have been used in cases such as the sintering of zirconia-hardened alumina nanocomposites, a process with temperatures up to 1300 °C. Microwaves resulted in an improvement of the nanocomposite microstructure as the alumina particle size decreased, increasing the zirconium content and thus a higher densification of these nanocomposites compared to the use of an electric furnace by means of the conduction mechanism [72,73]. Another process with ceramics, in this case with a domestic microwave oven, is in the drying of silica sludge [55]. This new method reduces their volume and mass, reducing the process cost. In polymer processing, Porto et al. [31] investigated with a domestic microwave oven, and benzoyl peroxide as a catalyst, the polymerisation process of various monomers (vinyl acetate, styrene, methyl methacrylate and acrylonitrile), which resulted in an efficient method.

2.2. Behaviour of matter with microwaves

Any material can be described electromagnetically by its relative dielectric permittivity, $\epsilon_r = \epsilon' - j\epsilon''$, composed of the dielectric constant (ϵ') which determines the spatial distribution of microwaves within matter, and the loss factor (ϵ'') primarily responsible for the conversion of microwaves into thermal energy in nonmagnetic materials [120]. For magnetic materials, the permeability (μ') and magnetic loss (μ'') determines the ability to store and convert the magnetic energy to heat [24]. These parameters are dependent on the operating frequency and temperature reached by the material. The power penetration depth, (D_p) is the distance from the surface of the sample at which the power decreases by a factor $1/e$, and this is dependent, not only on the dielectric and magnetic properties (ϵ' , ϵ'' , μ' , μ''), but also on the volume of the object, its density which affects these properties [116,117], and the incident wavelength (λ_0) [97,118].

$$D_p = \frac{\lambda_0}{2\pi\sqrt{2\epsilon'\sqrt{1+(\tan\delta_e)^2}-1}} \quad (6)$$

The electromagnetic power absorbed by a material, P , depends on the microwave radiation penetrating the material by:

$$P = 2\pi f\epsilon_0\epsilon''E_{rms}^2 + 2\pi f\mu_0\mu''H_{rms}^2 \quad (7)$$

where μ_0 is the magnetic permeability of air ($H\ m^{-1}$) and μ'' is the magnetic loss factor, negligible in non-magnetic materials. E_{rms} and H_{rms} are the root mean square of the electric and magnetic fields, respectively. The factors $2\pi fE_{rms}^2$ and $2\pi fH_{rms}^2$ are dependent only on the applicator and the emitted power. To obtain the energy absorbed by the material, Ayappa [121] obtained a critical thickness in slabs, which showed that valid power profiles are obtained using Lambert's Law, considering an exponential decay of the energy absorption. Therefore, this approximation is used for the calculation of power in samples with sufficiently thick section:

$$P = P_0e^{(-2\alpha y)} = P_0e^{(-2y)/\delta} = P_0e^{-y/D_p} \quad (8)$$

where P_0 is the incident power (W) at the surface of the sample, y is the distance inside the material and α is the attenuation factor ($dB\ m^{-1}$) which is the inverse of the characteristic penetration depth (δ), i.e. the distance at which the field strength decreases by the same factor as the power penetration depth [98,122]. Note that D_p is half of δ .

Therefore, not all materials interact in the same way with microwaves. As the electromagnetic waves penetrate into the material, the electric and magnetic field intensity vary [13]. In addition, as the material increases its temperature, these intensities are updated, making it difficult to measure the power absorbed. The time evolution and spatial distribution of the temperature can be calculated by applying the following energy balance [122]:

$$D_p = \frac{\lambda_0}{2\pi(2\epsilon'\mu')^{1/2}} \left[\left(1 + (\tan\delta_e)^2(\tan\delta_\mu)^2 + (\tan\delta_\mu)^2(\tan\delta_e)^2 \right)^{1/2} + \tan\delta_e\tan\delta_\mu - 1 \right]^{-1/2} \quad (5)$$

where $\tan\delta_e = \epsilon''/\epsilon'$ and $\tan\delta_\mu = \mu''/\mu'$ are the loss tangents.

Magnetite (Fe_3O_4) and some iron oxide based ceramics are materials which interact with electric and magnetic fields. In the case of the materials interacting only with the electric field the expression for the D_p reduces to:

$$\rho C_p \frac{\partial T}{\partial t} = 2\pi f\epsilon_0\epsilon''E_{rms}^2 + 2\pi f\mu_0\mu''H_{rms}^2 + k_T\nabla^2 T \quad (9)$$

where ρ is the density of the material (kg/m^3), C_p is its specific heat (W), T is the temperature distribution ($^{\circ}C$), t is the time (s), k_T is the thermal conductivity ($W/m^{\circ}C$).

According to the evolution of the electric and magnetic fields as they penetrate the different materials, they can be classified as follows:

- **Transparent:** the strength of the E and H fields hardly varies as they penetrate the material.
- **Absorber:** as microwaves advance inside the material E and H decreases as the ϵ'' increases. These microwaves are absorbed and converted into heat.
- **Reflective:** microwaves are reflected, with negligible penetration depth and therefore do not absorb microwave energy.

A last group would be mixed or composite absorbers, i.e., materials which are able to absorb just one microwave phase or those, that being composites, have one of their components microwave absorbers. This selective heating mode is very advantageous over traditional processing techniques [123,124]. For example, this is the case for fibre-reinforced PMCs, where this selective heating is used to generate heat at the joint location, without affecting the properties of the rest of the volume covered with a masking material [33]. Zhou et al. [37] addressed the problem to cure the metal-like carbon fibre reinforced polymer (CFRP) composite, as its laminates are highly reflective. By introducing a resonance structure composed of an 18 μm thick copper foil and a dielectric spacer into a resonance-insulator structure (RSI), the CFRP components achieved perfect absorption derived from the magnetic resonance between the RSI with the CFR component and microwaves. This configuration induced strong currents within the laminates and converted the microwave energy into Joule heat. Compared to conventional heating with autoclaves, a higher heating efficiency (36.3 times) and a 99.2% reduction in energy consumption were obtained.

Materials such as PTFE (Polytetrafluoroethylene) or quartz are considered transparent. These ones have a very low ϵ'' , with almost zero value and, therefore, a large D_p . Thus, quartz glass is a material widely used as sample container in microwave systems, with D_p up to 160 m [125].

Metallic materials have a high value of ϵ'' , so their D_p is very small, termed as skin depth (D_s) [126,127].

$$D_s = \frac{1}{\sqrt{\pi f \mu \sigma}} = 0.029(\rho \lambda_0) \quad (10)$$

According to the electronic structure of metals, they are characterized by the absence of the forbidden band. When microwaves are irradiated on a metal of considerable size, they induce a magnetic field that addresses free electrons to the surface sample. These electrons can accumulate in corners in clouds and produce discharges. In the design of microwave applicators, the material cavity walls are perfectly smooth, ensuring that the microwaves are reflected without heating. Zhou et al. [79], as in the case of microwave heating of metal-like CFRP laminates, used copper-clad polyimide films as electromagnetic resonators (EMRs) to heat metal plates of 5 materials, magnetic (iron, nickel and invar) and non-magnetic (tin and zinc). This metal-insulator-metal (MIM) configuration induced high currents in the sample that heated the plate by "resistance heating". Although these currents decay rapidly, as metallic materials have excellent thermal conduction, the samples were fully heated. The MIMs were successfully tested in a standard microwave heating process and in a soldering process. Microwave absorption and heating of metals is possible when a high temperature known as critical temperature (T_c), and ranged from 400 °C to 600 °C, is reached [65]. In the case of metal powders, their particle size can be comparable to D_s , which contributes to the volumetric and uniform heating of these nano-sized powders. Anklekar et al. [75] obtained improved mechanical and microstructure properties sintering copper and steel with microwave powder metallurgy.

2.2.1. Container material in microwave heating applications

When designing a microwave cavity for heating at moderate or high temperatures (from 500 °C and above), it is necessary to consider the container. The material container has to be microwave transparent with high mechanical and chemical resistance, have high durability to thermal shock, with special attention to hot spot formation. Common materials are PTFE, fused quartz or alumina-based materials. The following Table 3 lists the dielectric parameters of these low-loss materials.

Behrend et al. [129] investigated the feasibility of different microwave-transparent refractory materials for high-temperature applications. In the case of glass melting, the container material has to be stable at above 1177 °C which is the melting temperature of glass [63]. Experiments by Behrend et al. [129] consisted of heating corundum and boron nitride tubes containing oxidic materials and placing them in a single mode microwave applicator. The power was up to 3000 W until melting. Corundum was evaluated as an alumina-based ceramic material, transparent to microwaves, stable at high temperatures, and sufficiently resistant to thermal gradients. Boron nitride, a less known material, behaves as transparent to microwaves. Its dielectric properties vary from 1.5 to 2.5 for ϵ' and from almost zero to 0.05 for ϵ'' , in temperature range from room temperature to 1800 °C and frequencies 615 MHz and 1412 MHz [130]. These values were measured by the CPM [131]. In addition, this material results to be mechanically and chemically resistant. Boron nitride tubes proved to be more suitable for high-temperature microwave applications as it did not reveal cracks, unlike corundum tubes (Fig. 10), a refractory material that shows a large increase in ϵ'' at temperatures above 800 °C, depending on the purity of Al_2O_3 [132].

2.3. Microwave heating techniques

Since the interaction of materials with microwaves is nowadays better known, new systems are developed for different industrial sectors. In this way, Martin and Navarrete [21] presented a review of new advances in microwave systems in three major application areas such as the synthesis of composite materials and nano-materials, the processing of natural compounds and the intensification of catalytic reactions, which demonstrates the increasing relevance of this technology in the industry.

Many factors are involved in microwave-assisted heating: frequency, power, number of microwave ports, their position, geometry, material and movement of the sample [28,29]. A poor design means that a large part of the irradiated microwaves are not absorbed by the sample [133]. Considering these parameters and the heating mechanisms, different techniques are available to achieve efficient processes.

2.3.1. Direct heating

According to the classification of materials with respect to their microwave behaviour, microwave absorbers can be heated volumetrically by exposing them directly to microwaves. In this case, the effectiveness of this direct heating is based on the temperature uniformity.

Table 3
Dielectric properties of some low loss materials.

Material	Dielectric constant (ϵ')	Loss factor (ϵ'')	Refs
Fused Quartz (at 3 GHz, 25 °C)	3.800	0.00006	[128]
Polytetrafluoroethylene, PTFE *	2.066	0.0007	[110]
(at 2.1563 GHz, 22 °C)			
Corundum (at 2.45 GHz, 23 °C)	3.900	0.0012	[111]
Alumina (at 2.1393 GHz, 22 °C)	8.823	0.0007	[110]

* PTFE temperature resistance is up to 260 °C.

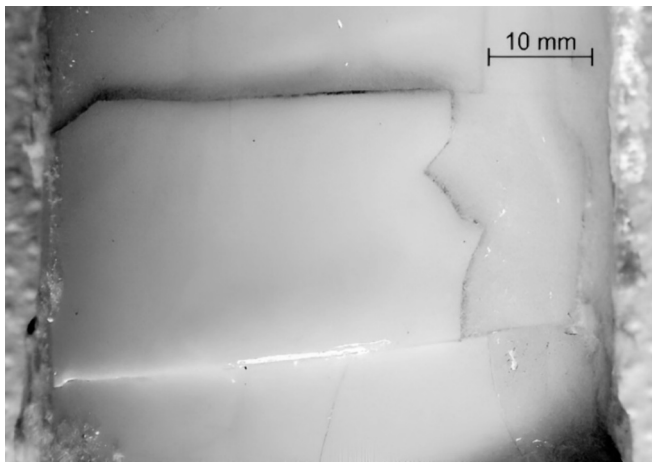


Fig. 10. Cracks in a corundum tube after microwave melting experiments [129].

The inhomogeneous distribution of the electromagnetic field can produce hot spots, which can lead to the formation of “thermal runaway”, where ϵ'' increases exponentially, resulting in uncontrollable temperature increases [134]. These thermal instabilities occur, for example, in some ceramic materials such as alumina, silica or zirconia due to a strong dependence of internal heat generation with temperature. According to Roussy et al. [135] there is a critical temperature at which the dielectric material cannot be heated uniformly because at some point the incremental absorbed power exceed the dissipated power. These thermal instabilities may result in non-uniform properties and cracking [134,136,137].

When a material is directly exposed to microwaves, depending on the type of material, different physical mechanisms lead to the heating of the material:

- In non-magnetic (diamagnetic or paramagnetic) materials only the electric field is involved, so the heating is due to:
 - o dipole losses, i.e., the agitation of the dipole molecules, since they reorient themselves to be in phase with the oscillating electric field
 - o conduction losses, or heat generated due to frictional, elastic or inertial forces, by pushing the electrons in the opposite direction to the external field, generate heat. It is what occurs with Cu, Al, Ni, Si or Fe, and MMCs.
- In magnetic (ferromagnetic) materials the magnetic component is also involved through hysteresis loss, electromagnetic eddy currents, or residual losses such as domain wall resonance or magnetic resonance.

2.3.2. Hybrid heating

This technique is used to heat composites with an absorbing material and another one that needs to reach its T_c to be able to absorb microwaves. When the composite material is exposed to microwaves, the part that can be heated increases its temperature, and, thanks to conduction and convection, heats the non-absorbing part until it reaches this T_c , then the entire volume is heated by microwaves [126]. With this technique it is possible to process materials such as metals [138]. Srinath et al. [139] used this technique with charcoal powder as a susceptor medium for the initial coupling of microwaves with stainless steel, which has a D_s of about 3.1 μm . Other main susceptors used in metal processing include silicon carbide, graphite, pure alumina or zirconia [140]. In addition, in metallurgy, this technique has been used as a sintering technique for metal-based materials such as aluminium, magnesium and lead-free solders. Compared to traditional resistance sintering, bidirectional sintering, i.e. direct heating of the powder

compacts and radiative heating of the silicon carbide susceptor, has given better tensile test results [93,141].

2.3.3. Selective heating

Selective heating is used when only certain part of the sample needs to be heated. In this case, the material or component that is not affected by the microwaves is coated with a masking material that reflects the microwaves [13]. Singh et al. [34] used this technique in the microwave-assisted joining process of “green” thermoplastic composites, resulting in an optimised process with reduced time and consumption requirements. In addition, it is widely used in microwave-assisted extraction of natural compounds. This is the case for the selective heating of biomass constituent components material in a sample with an absorbent and transparent phase mixture. [142]. Biomass easily absorb microwaves at room temperature as it contains moisture (water). This technique requires high power densities to allow that volumetric heating dominates over conventional heat transfer, enabling selective heating.

2.4. Temperature measurement methods

As in any thermal process, microwave heating control comes through measuring temperatures. To improve the microwave heating uniformity of a silicone rubber, Sun [143] implemented an active temperature control method. The tracking quality of the controlled temperature in five positions on the sample surface was of great influence to adjust the operating power of multiple microwave sources with grey-box (model based) and black-box (data-based) algorithms. Nevertheless, this reference control of the model is not valid with composites as they are a phase-changing type of material. In order to achieve significant improvement in temperature uniformity in microwave curing of PMCs, Zhou et al. [38] applied a Convolutional Neural Network (CNN) architecture to learn the dynamic heating pattern under a microwave control strategy. As many properties of PMC change as a function of the degree of cure, small discrete intervals were considered with constant properties in each interval. This control system was applied with a fibre optic fluorescence temperature measurement system with 20 probes installed in a heptagonal cavity with 21 microwave sources distributed on each side. Compared to the random superposition of unequal electromagnetic fields, the results showed a significant improvement. Additionally to common temperature measurement requirements (accuracy, sampling frequency and appropriate and reliable measuring points), microwave heating imposes to pay attention to the independence of the measurements from any background radiation and to avoid electromagnetic interference [144]. That is the reason why it is advisable to use different types of temperatures gauges, such as fibre-optic probes or thermocouples, together with a thermographic camera. In addition, a single measurement can lead to misinterpretations due to temperature inhomogeneities. Gangurde et al. [145] designed a customised microwave cavity with a dual thermal camera-thermocouple temperature measurement method to obtain real-time high temperature in catalytic reactors. This system was applied in a platinum-on-carbon catalytic bed for the dry reforming of methane. Due to the presence of different media and reactant gases and thus the multi-parametric dependence of the emissivity, a contact sensor was needed together with the camera. This system proved to be valid for detecting the generation of hot spots and continuous temperature monitoring. The following sections discuss some of the advances in the most commonly used temperature measurement techniques, which according to Grellinger and Janney [146] are thermocouples, fibre-optic sensors, pyrometers and thermographic cameras.

2.4.1. Contact method: Thermocouples and fibre-optic sensors

Knowing that thermocouples are based on metals, they may generate electromagnetic eddy currents giving erroneous readings, behave like antennas transmitting radiation to the outside, and alter the distribution

of the electromagnetic field [147]. In the manufacture of sand-casting moulds, in order to avoid arcing and microwave interference in temperature measurement, thermocouples were introduced into the heating zone shortly after the microwave oven was switched off. This is not possible in the majority of cases, where continuous, precise measurements are required while the sample is being heated. Yongguang et al. [66] proposed in zinc metallurgy to use an electromagnetically insulated thermocouple fixed with a special thin tape protective shield of aluminium. The measurements obtained were up to 800 °C with associated errors of ± 2 °C. A different way of measuring with contact on the sample, avoiding electromagnetic interference, is proposed by Arzhanikov et al. [148] using BeO ceramic probes with high thermal conductivity (270 W / m °C) that transfer heat by conduction to thermocouples located in areas without the presence of the electromagnetic field. In this case, the time needed to measure the actual temperature of the sample is about 30 s, which also limits their use in moving samples. There are several cases of using thermocouples protected with an earthed sheath to avoid induced currents. Bradshaw et al. [149] used K-type stainless steel coated thermocouples to measure temperature and power output control carbon regeneration for the lab-scale carbon-in-pulp process. To prevent arcing, the metal sheath was grounded to the cavity and the thermocouples were of the ungrounded tip variety. It was possible to reduce interference with thermocouple resolution (± 1.5 °C). The shielded extension cables were grounded with the thermocouple amplifier circuits, designed to exclude electromagnetic interference, as far away as possible from the high voltage area. Van de Voort et al. [150] and Ramaswamy et al. [151] used thermocouples shielded by an aluminium tube and grounded to the cavity wall, obtaining measurements with errors of less than ± 2 °C. However, whenever earthed shielded thermocouples are used, connection to a signal conditioning and data acquisition unit located outside the cavity is required, which is inconvenient for measurements of moving samples in continuous microwave heating [152]. An alternative is using so-called wireless sensors. Devices containing together the data logger and the sensor probe [101,153–155]. As a disadvantage of these sensors, their size may limit them to measurements at a single point within the sample. With a cylindrical shape - diameter of 1.5 cm and length of 2.2 cm - where the data logger is located, and the probe - length of 5 cm - preliminary experiments or numerical simulation are required to determine the heating pattern, and consequently position the sensor. These sensors must be positioned perpendicular to the electric field vector, otherwise local overheating will occur as a result of an intense singularity [154]. This is the reason why their use in multimode cavities is excluded.

Fibre-optic sensors are presented as an alternative to thermocouples as they are not susceptible to electromagnetic interference. Despite their high measurement speed and high accuracy, with errors of ± 0.1 °C, they have limitations such as a reduced measurement range, up to 300 °C. In turn, as is the case with shielded thermocouples, the movement of the sample is limited. The optical fibre, in contact with the sample, is connected to a signal processor outside the cavity [152]. In addition, they are fragile and expensive [156,157]. Their technology is based on the temperature-dependent light scattering [158], like Raman scattering is. Temperature-reactive components (phosphors or liquid crystal) are integrated into the tip of the flexible fibre, and the optical fibre transmits the light from a source, e.g. LEDs emitting light pulses. Among the technology used, interferometry (Bragg gratings, Fabry-Perot resonator) or fluorescence are the most remarkable. The signal conditioner is able to detect changes in backscattered light (Rayleigh scattering). In addition, the lower the temperature at the sensor tip, the longer the light delay time [158–160]. Ramopoulos et al. [35] used a fibre-optic thermocouple for temperature measurements in dielectric characterisation as part of the industrial application of continuous microwave depolymerisation of polyethylene terephthalate (PET). This sensor, type Optocon Fotemp-H with the outer shell of PTFE and a GaAs crystal fixed on its tip, was placed at the centre of the sample in a dual-mode cylindrical cavity. The TM₀₁₀ mode provided a homogeneous sample heating.

These measurements were used by a PID control algorithms to control the temperature along a pre-set value up to 180 °C. This fibre-optic sensor was a success for this application, providing high-speed measurements with high accuracy and precision due to the dependence of the semiconductor's forbidden band position on temperature. Ano et al. [161] demonstrated in situ measurement of local temperatures in chemical reactions and materials science. In these experiments, photoluminescent probes were positioned in nano-spaces of these two nano-hybrid systems: BaTiO₃ particles coated with a SiO₂ layer (BaTiO₃-SiO₂), and tungstate particles coated with embedded ammonium cations (C₁₆N⁺-W₂O₇²⁻). This method proved to be an important tool to investigate the mechanism of the special effects of microwaves. Finally, it is important to highlight its relevance in fast reaction processes. Bacsa, et al. [162] evaluated the suitability of a fast response fibre optic probe system in the preparation of high purity peptides on Tentagel or ChemMatrix resin in a short time. This was of great relevance for adjusting the power input, feeding the magnetron control system with the measurements obtained.

2.4.2. Non-contact method: Pyrometers and thermal imaging cameras

The operation of this measuring equipment is based on the detection of the infrared signal emitted by bodies as a function of their temperature. The intensity or spectral emittance of the black body as a perfect absorber of radiation is based on Planck's law. This law is usually represented in a series of curves, Fig. 11, which represent the energy density at different wavelengths and temperatures of the body [163]. This emission of infrared radiation in materials differs from the ideal black body. Its emissivity describes the radiated intensity of the material relative to that of the black body. Its value is always < 1 , and depends on the viewing angle, wavelength, temperature, surface structure of the material, its properties, among others. In some cases, it can be assumed that this emissivity value is constant for all wavelengths, known as grey body approximation. For higher temperatures (from 300 °C) the emissivity varies more strongly, which makes it necessary to use emissivity curves. These curves are very often not available, which makes it necessary to obtain them experimentally with another method of measuring their temperatures [16]. Its main advantage is the ability to measure temperature remotely, without the need for contact. In multimode applicators it is of great interest as any slight interference in the cavity can lead to significant changes in the heating pattern [164]. These

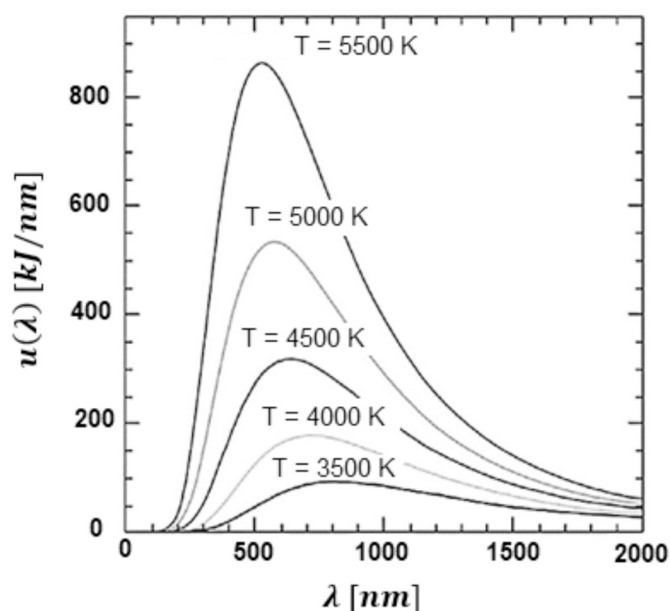


Fig. 11. Planck's Law blackbody radiation spectrum. Energy intensity curves (u) as a function of wavelength (λ) for different temperatures (T) [163].

devices are placed outside the microwave chamber and measured through an orifice. To avoid leakage of electromagnetic radiation, optical windows are used. Therefore, the measuring device is calibrated for the minimum reduction of infrared transmission that may be caused by this window, which may be constructed of quartz glass with a wired network to avoid microwave leakage. The field of view (FOV) of these devices is limited to the surface of the observed object, which does not make them valid for measuring temperatures inside matter. A distinction is made between the measuring devices for the detection of infrared radiation, according to the spatial resolution they provide: pyrometers and thermal imaging cameras. While pyrometers cover a small measurement spot of several millimetres, thermal imaging cameras provide a temperature map of the object's surface based on the grouping of radiation detectors (CCD arrays).

Pyrometers measure the average temperature of the observation point or area. Therefore, the larger the field of observation, the greater the error in the measurement. This is why their use is limited to the control of homogeneous temperatures, or predefined critical points, although it is worth mentioning the study conducted by Protasov [165], who developed an alternative to thermographic cameras to obtain the temperatures of the entire area of the sample. This is a program that allows reconstructing the temperature distribution image from the measurements obtained by pyrometers in a previously defined grid. Together with an interpolation model, a good fit in the reconstruction of the temperature field was achieved. Nevertheless, applications with microwave-assisted heating make use of the pyrometer to measure the process temperature at a single set point. Amini et al. [166] used a multimode microwave cavity with 1050 W power together with hydrogen as a reducing agent to decrease the number of carbonaceous materials in the reduction of magnetite. A pyrometer with a measuring range from 330 °C to 1500 °C is integrated into this system. Mondal et al. [76] also makes use of a pyrometer in the temperature measurement of a metallic particulate compact, in this case in a 6 kW 2.45 GHz multimode microwave furnace. The emissivity varies little with respect to temperature, so the emissivity was considered constant. It was shown that copper powder compacts absorb microwaves and can heat up to high temperatures, up to 1000 °C, more rapidly as the particle size and green density decreases. Huang [57] installed a pyrometer in a domestic microwave oven with a 600 W rotating plate to measure the temperature of beef sausages 75 °C to 80 °C. Through these measurements, and with an all-or-nothing control of the microwave power, the set point temperature could be maintained and the ability to inactivate *Listeria monocytogenes* bacteria was demonstrated. There are more examples of pyrometer use in microwave-assisted processes, for example in the characterisation of dielectric properties in a cylindrical cavity [110], which provides information relative to its use.

Thermal imaging cameras, unlike the pyrometer, are suitable for applications where the entire surface of the sample needs to be measured. Accurate measurement of the temperature distribution is more dependent on the emissivity invariance along the surface [167,168]. Instantaneously, in a few milliseconds, these cameras collect a broad spectrum of radiation in certain wavelength ranges [169]. The detected radiation can have various sources such as from the environment or the atmosphere between the sensor and the object to be measured [170]. To obtain values with resolutions of 0.05 °C, the temperature of the object to be measured must differ by at least 50 °C from the room temperature [168]. Thermal imaging cameras have been integrated into microwave systems for automated temperature monitoring and control, e.g. in the food industry for the preparation of dried fruits by continuously applying temperatures up to 80 °C [58], or the processing of PMCs such as polylactide acid (PLA) with graphene nanoplatelets (GNP) as strong microwave absorbers, with more rapid heating, up to 250 °C, as the GNP filler loading is increased to 4 and 8% [36].

2.5. Numerical simulation

With the evolution of microwave applications in industry, mainly in materials processing, numerical modelling has become a fundamental part of the design of microwave-assisted heating systems, with the ability to predict and control the variables necessary to achieve an efficient process. The numerical methods commonly used in these simulations are the finite difference time domain method (FDTD) and the finite element method (FEM). These methods are applicable in multiphysics simulation software such as COMSOL Multiphysics, CST Studio Suite, ANSYS HFSS or QUICKWAVE. These tools allow the creation or import of models. The validation of simulated results with those obtained by experimentation depends on the accuracy of the materials properties, and on the simplifications and boundary conditions assumed. The main properties to include in the simulations are the dielectric permittivity (ϵ), thermal properties such as conductivity (κ) and heat capacity (C_p), electrical conductivity (σ), or density (ρ). These properties are temperature dependent, which results in a non-linear and complex model that requires iteration of the different physical modules. In order to solve the multiphysics problem it is necessary to assume appropriate boundary conditions, like that the metallic walls of the system are perfect conductors, which implies that the tangential component of the electric field is zero. It is used for lossless metallic surfaces or as a symmetry boundary condition. Finally, the mesh is a fundamental part of any numerical simulation. Its size and shape affects the computational memory and, therefore, the time needed in the simulation, as well as the validity of its results. In electromagnetic problems, a maximum mesh size (S_{max}) is established based on the Nyquist criterion [171].

$$S_{max} < \frac{\lambda}{2} = \frac{c}{2f\sqrt{\epsilon'\mu'}} \quad (11)$$

where λ is the wavelength (m), f is the frequency (Hz), c is the speed of light in a vacuum (m/s), ϵ' is the dielectric constant and μ' is the relative permeability. However, Zhang et al. [59] suggest that using six grids per wavelength is preferable for the finite element solution of the Maxwell equations of electromagnetic distribution.

Among the latest publications based on numerical simulation for microwave heating, it is worth mentioning the contribution by He et al. [172]. In this work, the process of a new multimode cavity design with rotating waveguides was simulated. This design results in a rotating radiation structure. The finite element software used was COMSOL Multiphysics. The simulation results were validated by experimentation with different materials, from potatoes to pine wood, in different positions inside the cavity and with different shapes of the heated object. It was demonstrated that the proposed design is able to produce efficient and uniform heating of different materials with different dielectric properties, as the system adapts by changing the heating pattern, i.e. angle of the waveguides and heating time of each angle. There are several works where numerical simulation is used to validate new designs. Zhang et al. [173] studied the influence of the carrier tube on the heating of distilled water flowing inside a domestic microwave oven. The simulations were performed on models with different diameters of the carrier tube, with helical and straight tube, in the case of helical with different sizes of the circumference formed by the tube, and different number of turns. The simulation results were validated by experimentation, and the influence of helical tubes on the uniform heating of fluids was demonstrated. The mentioned works are performed within the food sector, operating at low temperatures up to 80 °C. Topcam and Erdogdu [49] worked on a similar concept in cylindrical and rectangular cavities to analyse the influence of the position and orientation of the fluid-carrying tube. In addition to simulating microwave-assisted processes for the food industry, due to the capability of these multi-physics packages, numerical modelling has been used for mining applications as a pre-process to facilitate the extraction of minerals with a consequent decrease in energy consumption [174]. Another application where

numerical simulation can be found in the metallurgical industry. For example, for joining dissimilar metals, using copper powder as an intermediate layer in a hybrid microwave heating process with temperatures up to $\sim 1060^\circ\text{C}$ [78].

The development of new microwave systems in the processing of composite materials involves the application of numerical simulation tools capable of predicting the evolution of the temperature depending on the properties of each phase that affect the electric and magnetic fields during heating. The SIMUTOOL project, within the European Research and Innovation Programme Horizon 2020, arises from the need to simulate the processing of composite materials by microwave heating with the aim of achieving advanced materials with lower electricity consumption for their processing [175]. The figure below, Fig. 12, shows the electric field distribution of a composite heated using the VHM (Vötsch Hephaistos Microwave) microwave.

3. Future perspectives of microwave heating

Microwaves technology has proved to be an outstanding alternative in different applications contributing to a circular economy. As more is known about how the different parameters involved in any microwave system interact, more and more advances are being made. To date, however, applications remain limited to the laboratory scale due to the complexity of the multi-physics process and poor reproducibility, as most processes are highly influenced by the electromagnetic, thermal and chemical characteristics of the material to be heated, which can result in thermal runaway, the position of the microwave source in the system, the input power, among others. There are several researching lines open such as the employment of less conventional frequencies and use of new-generation solid state generators in the scaling-up of microwave reactor designs [18]. Olszewska-Placha et al. [176] presented a combined active-passive methodology for the design of solid-state microwave ovens, in line with the need for new design formulas to maximise the benefits of solid-state technology. As seen previously in 2.3.2, the processing of materials that are a priori transparent or reflect microwaves have been successfully heated with this technology using susceptors, which in turn has improved the uniformity of the process in various potential applications from ceramic sintering to metal processing [24]. However, there are still issues to be resolved as the development of novel susceptor materials. A study is needed of the factors involved in the selection of a particular receptor, especially the shape

(powder, rod or tube) and positioning of the receptor with respect to the sample and extend this to various hybrid configurations. Mishra et al. [13] highlighted advanced materials processing as an arising area in microwave application, identifying research opportunities in improving microwave efficiency, improving tools for *E*- and *H*-field control, numerical simulation for better understanding of the physics involved, and micromachining techniques. With regard to the numerical simulation of microwave processes, Birla and Pitchai [177] suggest that the development of new systems depends on the development of faster and more refined numerical methods. Together with a better description of the complex permittivity of more materials, this will lead to faster, more controllable, and therefore optimised design processes. Finally, the combination of microwaves with other technologies creates a wide range of possibilities in industry. This is the case, e.g., of the combination of microwaves with ultrasound, which is of great interest in the chemical industry by combining the volumetric and selective heating of microwaves with the production of radical active species and the intensification of mass transfer and surface effects [178]. For its applicability at any scale, either laboratory or industrial, it is necessary to develop mathematical models that calculate in a single program the results in the process or reaction of interest. Following the same line of combining microwaves with other technologies, Mohamad Aziz et al. [179] present the combination of microwaves with other technologies (acoustic cavitation, vacuum, ionic solvent and a supercritical/subcritical approach) for the production of biodiesel and biolubricants by transesterification from vegetable oils or animal fats. Although the use of microwaves alone saves 60% of the operating time compared to conventional heating, there are drawbacks that are intended to be solved by their combination with other technologies. These are the inhomogeneous distribution of the electromagnetic field and thus of its temperatures, the insufficient penetration depth of microwaves and the reduced mass transfer efficiency. The following diagram, Fig. 13, summarises some of the future possibilities for microwave heating.

4. Conclusions

Microwave heating has great advantages over conventional heating. There are different applications where microwaves are implemented, from food industry to synthesis of composite materials, processing of different materials, or intensification of catalytic reactions. The capacity to directly transform energy into heat within the material, microwave heating results in faster and more controllable processes, producing, in many cases, improved product properties, as well as being able to connect the sources to clean energy, which leads to sustainable processes. In order to implement this heating method, a customised design is required for each application, which involves studying the applicator to be used, from single mode with limited sample quantity or multimode, where the uniformity of the heating can be affected. With regard to the materials to be processed, their heating is determined by their dielectric characterisation, which determines the electromagnetic power absorbed and transformed within the material. Depending on the behaviour of each material with respect to electromagnetic waves, it can be transparent, absorbent or reflective. A final group is comprised of composite materials. According to the material and the requirements of each application, the material can be directly exposed to microwaves, where the physical mechanisms that affect and cause its heating are different depending on whether the material is magnetic or not (contribution of the electric and magnetic field, or only electric). There are also techniques such as hybrid heating to heat materials that a priori are not susceptible to microwave heating, or selective heating, where only one phase or a specific area of a composite material needs to be heated. In parallel, the design of the system depends on its constituent materials. Several investigations focus on the ideal material to contain the sample to be heated. It must be transparent to microwaves and highly resistant to thermal shock due to the formation of hot spots. In addition, there are several applications where the main advantage of using microwaves is

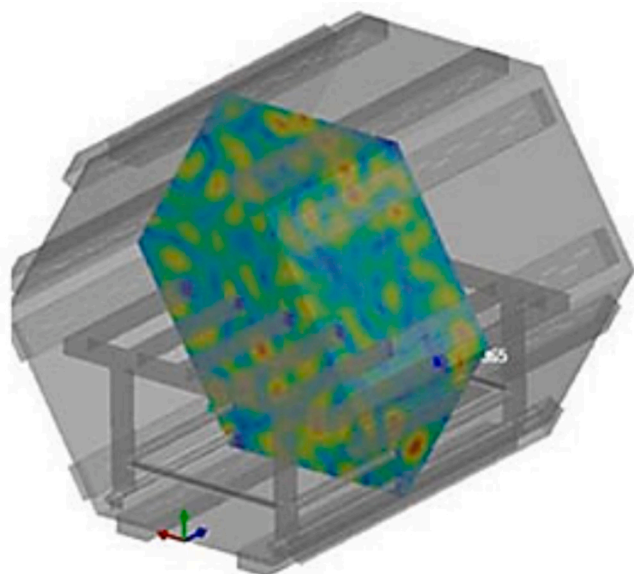


Fig. 12. Simulation of the electric field distribution inside the VHM microwave [175].

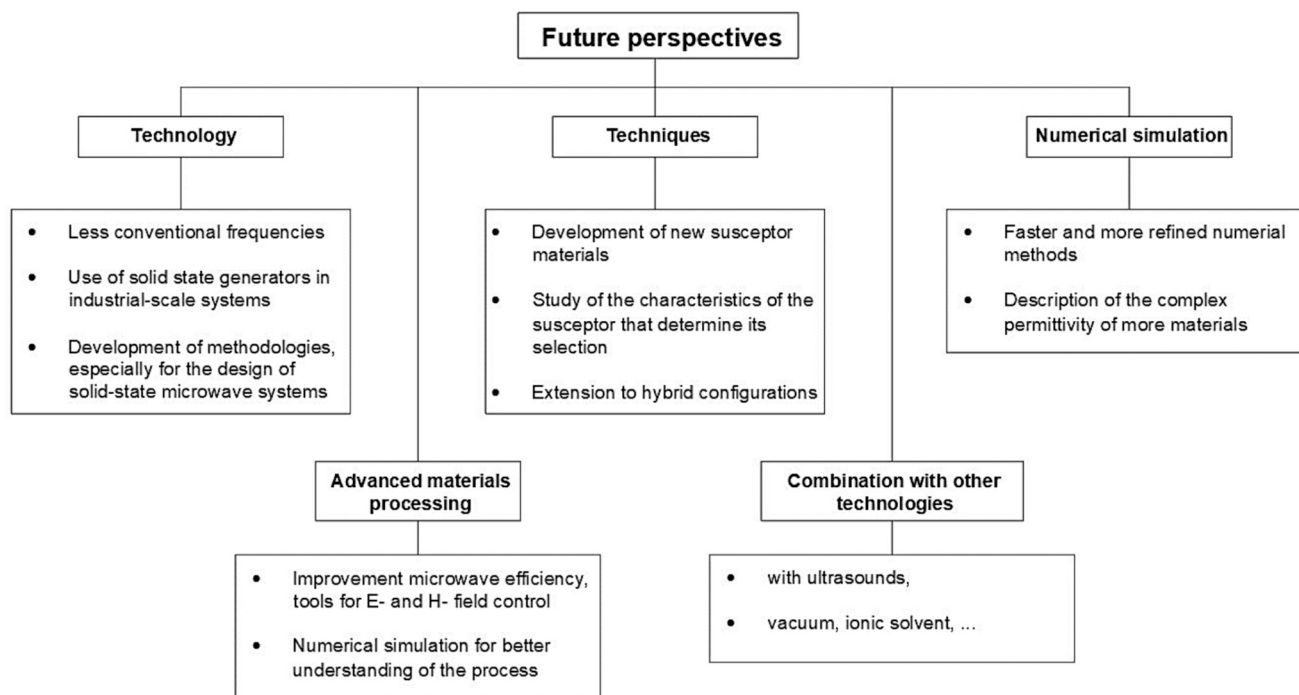


Fig. 13. Schematic of future perspectives for microwave heating.

based on the uniformity of the heating. For this purpose, a control system is required, which demands suitable measuring instruments for each process. Depending on the microwave applicator, temperatures reached, measuring speed, sample conditions (solid, liquid, static or moving), instruments such as metal or fibre-optic thermocouples, remote infrared sensors, or a combination of these can be used. Finally, numerical simulation, capable of predicting the effectiveness of microwave heating, emerges as a fundamental tool for the design of an efficient system for advanced materials processing.

CRedit authorship contribution statement

Cristóbal Valverde: Writing – original draft, Visualization, Investigation, Formal analysis. **Margarita-Manuela Rodríguez-García:** Writing – review & editing, Conceptualization. **Esther Rojas:** Writing – review & editing, Supervision. **Rocío Bayón:** Writing – review & editing, Supervision.

Declaration of competing interest

None.

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