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20 years of microwave technology developments using a coaxial antenna: From human health to green chemistry applications☆

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ABSTRACT

The global interest on microwave assisted chemistry (MAC) is due to the important benefits for the sustainable growth of green chemical industries and environmentally friendly progress of society. MAC has been firstly developed using oven-type microwaves (MWs) assisted reactors, which requires difficult and expensive industrial scale-up. In 2002, the development of coaxial dipole antenna allowed a direct application of MWs *in situ* in the reaction media, opening a crucial, novel versatile technological solution, making MW-assisted processes feasible in any configuration at any industrial level. Here, we present an overview of the technological development of 20 years research using a coaxial MW antenna for green chemistry and human health applications. The major MW technology breakthroughs described in these short-review are: i) MW-induced thermoablation machine, ii) *in situ* MW heating in open glassware chemical reactors, iii) electrodeless MW/ultraviolet (UV) lamps and photoreactors, iv) MW-high pressure reactor and v) solventless/simultaneous MW/UV/ultrasound (US) configurations. Applications for the synthesis of nanocatalysts, nanoparticles and polymers, advanced oxidative decomposition photochemical processes, solvothermal extraction of valuable products and biomass processing are discussed. Remarks on the scaling up of the extraction processes and frontier applications addressed to the treatment of current and future outbreak pandemic emergences are also shown.

1. Introduction

MAC is currently recognized as a standard method in the research and development of organic chemistry, green chemistry, food chemistry, material sciences, production of alternative energies and so on [1]. The global interest for educational, scientific, and industrial applications of MAC is due to its invaluable benefits for the sustainable growth of green chemical industries and environmentally friendly progress of society. MAC enables the production of products with the same quality than those obtained by conventional techniques, with higher heating efficiency, faster heating rate, lower operating costs, and lower environmental impact. Besides, the selective heating, superheating, precise control of temperature, solvent free reactions, ultrafast processing, and other remarkable advantages make MAC a unique and highly attractive technology [1].

MAC began at the mid-1980s as a curiosity driven research of chemists who used the commercial MW kitchen ovens as MW reactors. The technology was developed following the close cavity configuration and, during the 1990s and early 2000s, the first commercial oven-type MW reactors were available [1].

However, one of the main issues related to oven-type MW reactors in MAC deals with the investment cost required for their industrial scale-up along with a low penetration depth of MWs at 2.45 GHz, and a non-uniform distribution of the electromagnetic fields [2]. Alternatively, the coaxial MW technology, which uses a coaxial dipole antenna to apply the electromagnetic energy inside the reacting medium, is highly flexible and easily controllable, allowing a versatile scale-up through different reactor geometries. Moreover, the absence of dimensional constraints and the wide range of materials eligible as sample holder (teflon, quartz, glass, plastic and so on) make its industrial scale-up simpler.

We present below an overview of the technological development of 20 years research using a coaxial MW antenna for green chemistry and human health applications. Fig. 1 shows the timeline of the most decisive

☆ A beloved tribute to Iginio Longo, pioneer scientist of coaxial MW, for many of us mentor and friend.

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Abbreviations

MAC:	microwave assisted chemistry
MW:	microwave
US:	ultrasound
UV:	ultraviolet
EO:	essential oil
CDMEL:	coaxial electrodeless MW/UV lamp
PHMB:	p-hydroxymercurybenzoate
IONs:	iron oxide nanoparticles
MAE:	MW-assisted extraction
MWHD:	coaxial MW assisted hydrodistillation
SMWAE:	solventless MW-assisted extraction
US-MWHD:	simultaneous US/MW-assisted hydrodistillation

landmarks of the coaxial MW technology, patented by the physicist Iginio Longo ("Guglielmo Marconi" 2011 award from the Italian Physics Society) [3] who successfully applied coaxial MW antenna in many areas. In 2002s, the successful story began with the first breakthrough, a MW-induced thermoablation machine used in oncology [4]. First, applications on green chemistry, namely biomass processing, nanocatalysts, nanoparticles and polymer syntheses using ordinary open glassware reactors were approached. However, the versatility of the coaxial MW antenna led the development of novel configurations, including the photochemical reactor with the simultaneous MW and UV irradiation, the high-pressure high temperature MW reactor, the simultaneous US and MW reactor and, the solventless extractor. Applications on environmental remediation, extraction and biomass processing using the mentioned novel configurations are presented and discussed. The scaling up of extraction processes and recent frontier applications and technological developments aimed at managing current and future pandemic outbreaks are also shown, along with several remarks on these applications.

2. *In situ* MW heating in ordinary open glassware reactors

The *in situ* application of MW power using the coaxial dipole antenna technology is a safe and efficient method for the activation of chemical processes [5].

Open-end coaxial MW antenna is an extremely versatile tool of general interest in chemistry, enabling fast heating in ordinary open glassware, in closed metal vessels at high pressures, in batch or in continuous flow conditions. This technology instead of a closed oven gives the operator the possibility of monitoring and controlling a variety of chemical physical processes, with practical and economic advantages [5] (Fig. 2-a). It is possible to use several of these MW antennas in newly designed or already available large volume glass or metal reactors useful at industrial scale.

Gentili et al. designed a very efficient cap-choked MW antenna to directly heat a liquid medium. The metallic choke blocks the propagation of the reflecting wave that turns back towards the MW generator [6].

The MW antenna was used for the *in situ* activation of the synthesis of ruthenium, palladium or silver nanocatalysts, allowing to develop greener nanosynthesis processes characterized by shorter reaction times, reduced energy consumptions, better yields and with the possibility of easy scale-up [7].

The MW antenna showed great potential also in the synthesis of optical whispering gallery mode micro resonators by a single-step time efficient MW-assisted heterogenous polymerisation (Fig. 2-b1) [8]. Another interesting application was the synthesis of silver nanoparticles using rosemary essential oil (EO) as a renewable reducing agent (Fig. 2-b2). The coaxial dipole antenna allowed to promote both rosemary EO extraction and silver nanoparticles synthesis in aqueous solutions at atmospheric pressure [9].

Recently, the MW-coaxial antenna technology was employed in the (i) hydrodistillation of clove buds to extract essential oils and polyphenols (Fig. 2-b3) showing that the lignocellulosic solid residue obtained by MW extraction is richer in cellulose/hemicellulose than in conventional hydrodistillation [10], and (ii) keratin extraction from poultry feathers (Fig. 2-b4), obtaining higher protein yields with respect to conventional heating mode [11]. In this last application keratin was extracted in a one-pot process and used without further purification in the electrospinning process to produce protein based-bioplastics [11].

3. Photochemical processes: simultaneous MW and UV irradiation in an integrated novel photoreactor

The simultaneous application of MW power and UV light leads to more effective photochemical processes [12]. The MW/UV irradiation can be done in commercial MW oven or in a waveguide applicator using a mercury vapor lamp but, from a practical point of view, this configuration has several drawbacks, particularly the lack of control of the MW power which causes low efficiency of the MW/UV process [12]. The coaxial electrodeless MW/UV lamp (CDMEL) (Fig. 3a) developed by Ferrari et al. [13] in which the optical radiation emitted by the gaseous plasma discharge is produced by the coaxial dipole antenna, allows to measure, and control the MW power, leading the investigation of the energetic features of the process [13].

The lamp was successful employed to produce UV radiation and volumetric heating, contributing to oxidative decomposition of Acid Orange 7 in aqueous hydrogen peroxide solution [12]. Angeli et al. proposed the MW/UV method for the quantitative conversion in continuous flow of p-hydroxymercurybenzoate (PHMB) and thiol-PHMB complexes to Hg(II), without using chemical oxidizing reagents, avoiding the use of toxic carcinogenic compounds and obtaining a yield significantly higher than that obtained with a commercial UV lamp [14]. The oxidative decomposition of atrazine in water in presence of hydrogen peroxide [15], and the oxidative bleaching of Rhodamine B dye gave also interesting results. In the latter case the apparent activation energy of the bleaching process (Fig. 3-b1) was determined. These results proved that MW/UV photoreactor is suitable for the pretreatments of wastewater [16].

The photoreactor was tested in the detemplation of mesoporous materials (MCM-41), as alternative method to the US/NH_4NO_3//MeOH surfactant extraction [17].

Recently, CDMEL system has been employed in AirSterizUv project to develop an innovative technology to produce ultraviolet radiation at the wavelength $\lambda = 253$ nm aimed to sterilize the conditional air of public buildings, to reduce the infection risk from SARS-Cov-2 and/or other future threats (Fig. 3-b2).

4. MW-assisted solvothermal processes using a high-pressure MW-assisted reactor

The versatility of the MW-coaxial antenna technology led to its implementation in a high-pressure high-temperature stainless steel reactor allowing to significantly reduce the time and the cost of the conventional solvothermal processes (Fig. 4-a). Thanks to a versatile control approach, the system can operate isothermally, when the MW power applied is regulated to keep constant the medium temperature, or adiabatically when the MW power applied is kept constant. The reactor was designed for the hydrothermal heterogeneous catalytic decomposition of microcrystalline cellulose using zeolite beta as catalyst (Fig. 4-b1) [18]. The use of a MW reactor and a heterogeneous catalyst in different hydrothermal operating conditions (up to 210 °C and 18 bar) increase the product yields and decrease reaction time respect to conventional heating. Zeolites interact with MW, decreasing the energy consumption and the heating time. The MW-assisted high-pressure reactor was also successfully employed for the solvothermal syntheses of iron oxide

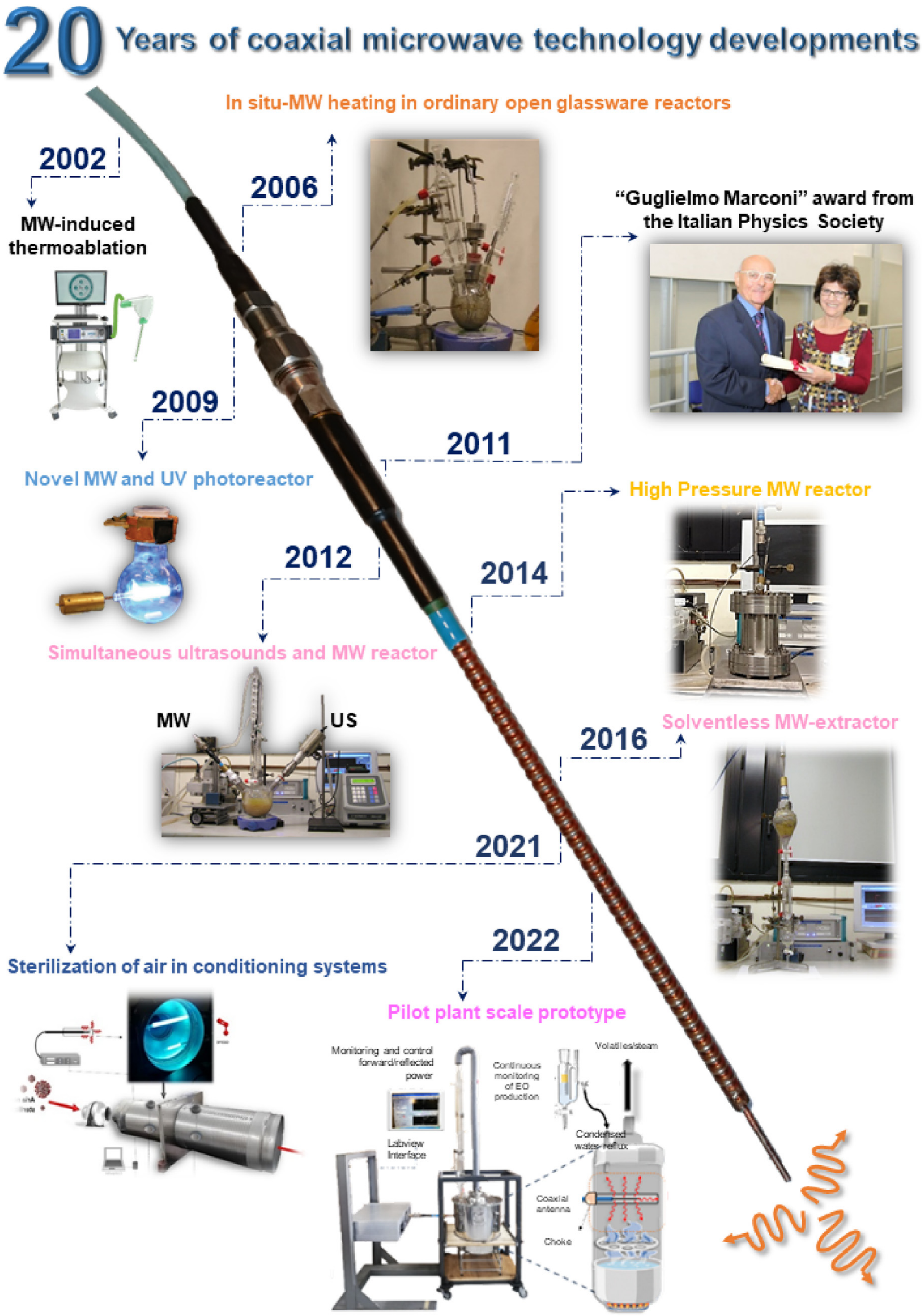

Fig. 1. Chronological timeline of major breakthroughs of the coaxial MW technology developments in green chemistry and human health applications. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

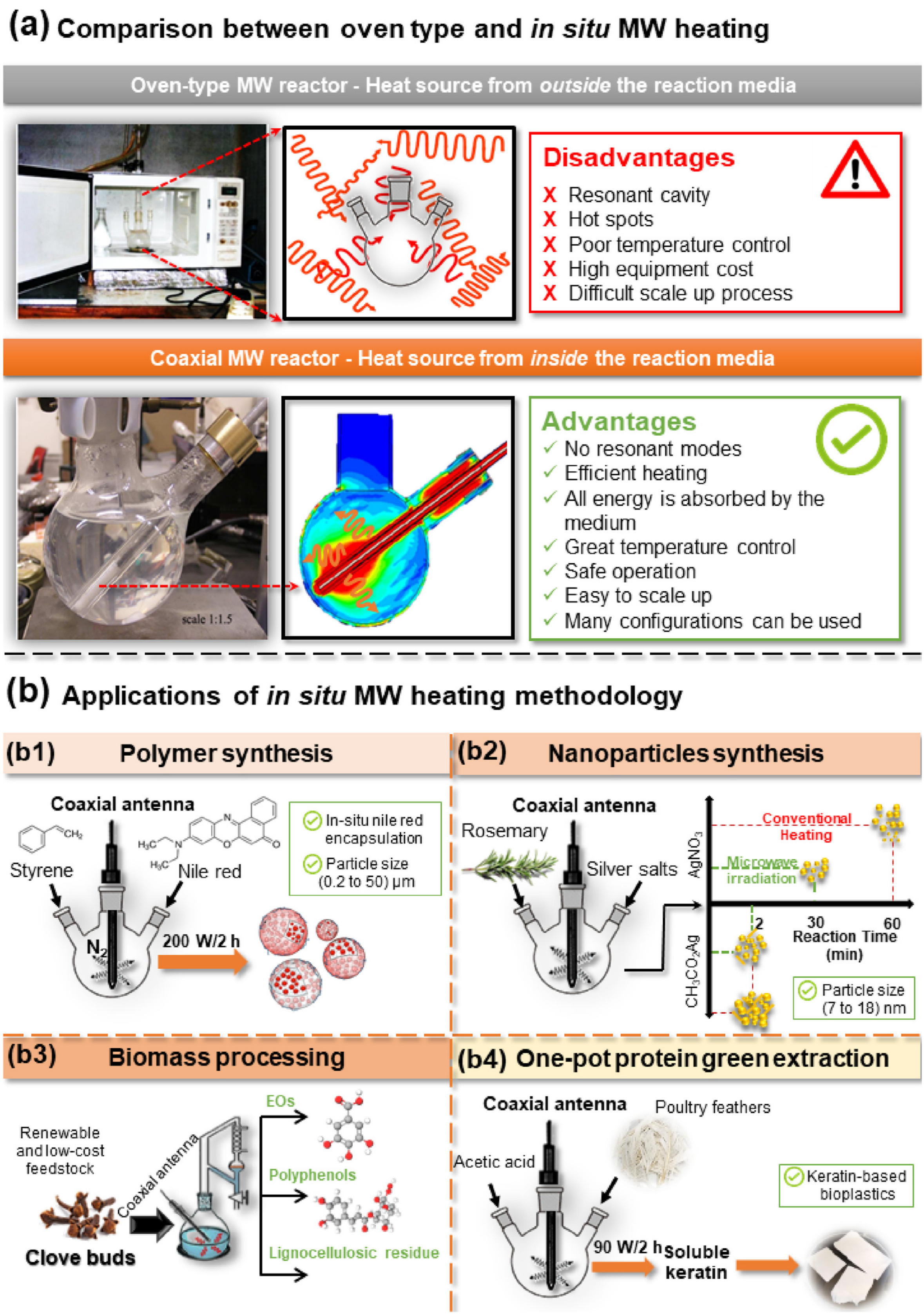

Fig. 2. (a) Comparison between MW oven *vs* coaxial antenna reactors with its electric field distribution. (b) MW heating in open glassware chemical reactors for the (b1) synthesis of optical whispering gallery mode microresonators, (b2) synthesis of silver nanoparticles using rosemary EO, (b3) hydrodistillation of clove buds to extract EOs, polyphenols and lignocellulosic compounds, (b4) keratin extraction from poultry feathers.

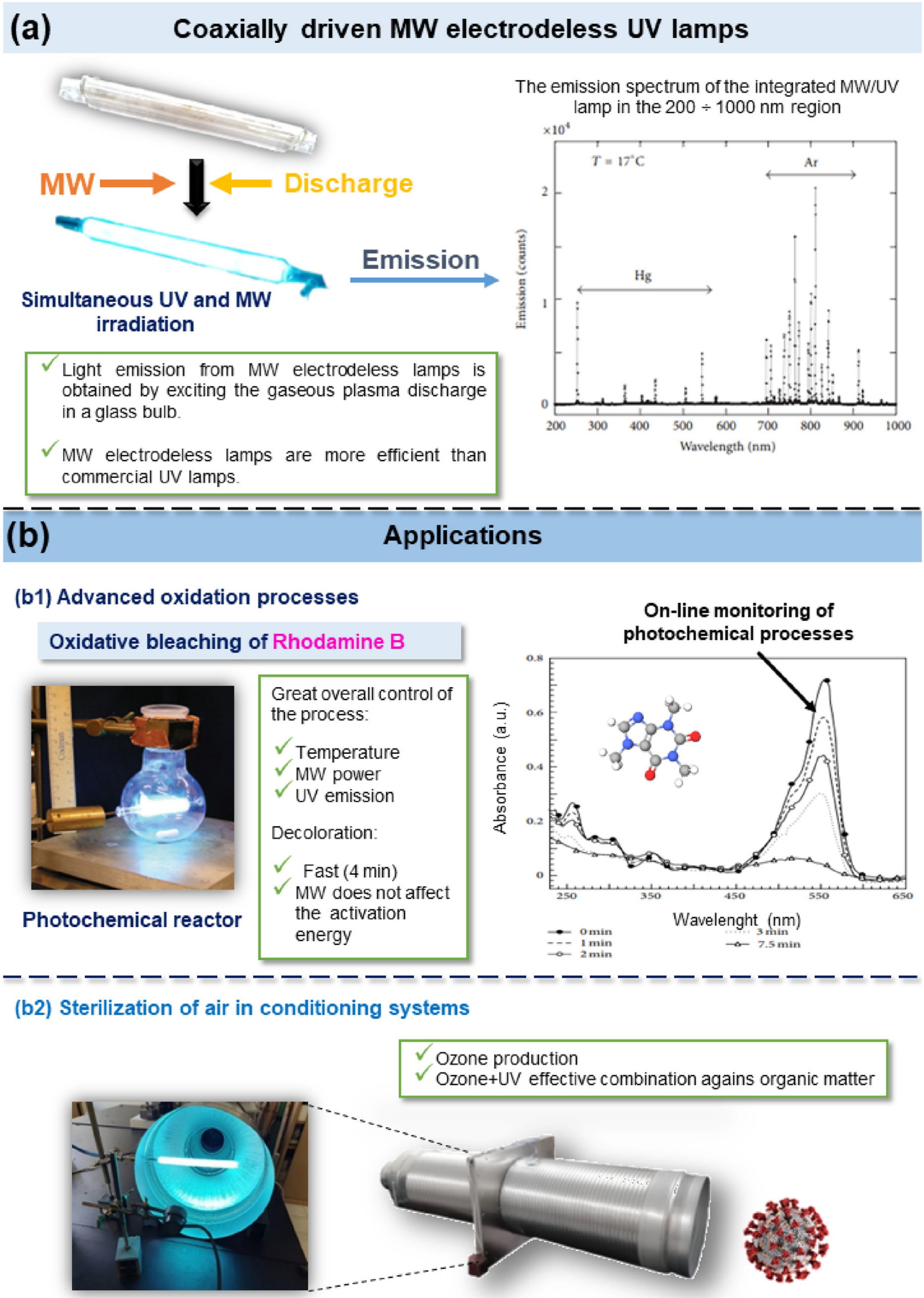

Fig. 3. (a) Coaxial electrodeless MW/UV lamp. (b) Application of MW/UV lamp for the: (b1) oxidative bleaching of Rhodamine B dye, (b2) Sterilization of air in conditioning systems (AirSterizUv project, http://www.iccom.cnr.it/en/airsterizuv-2/).

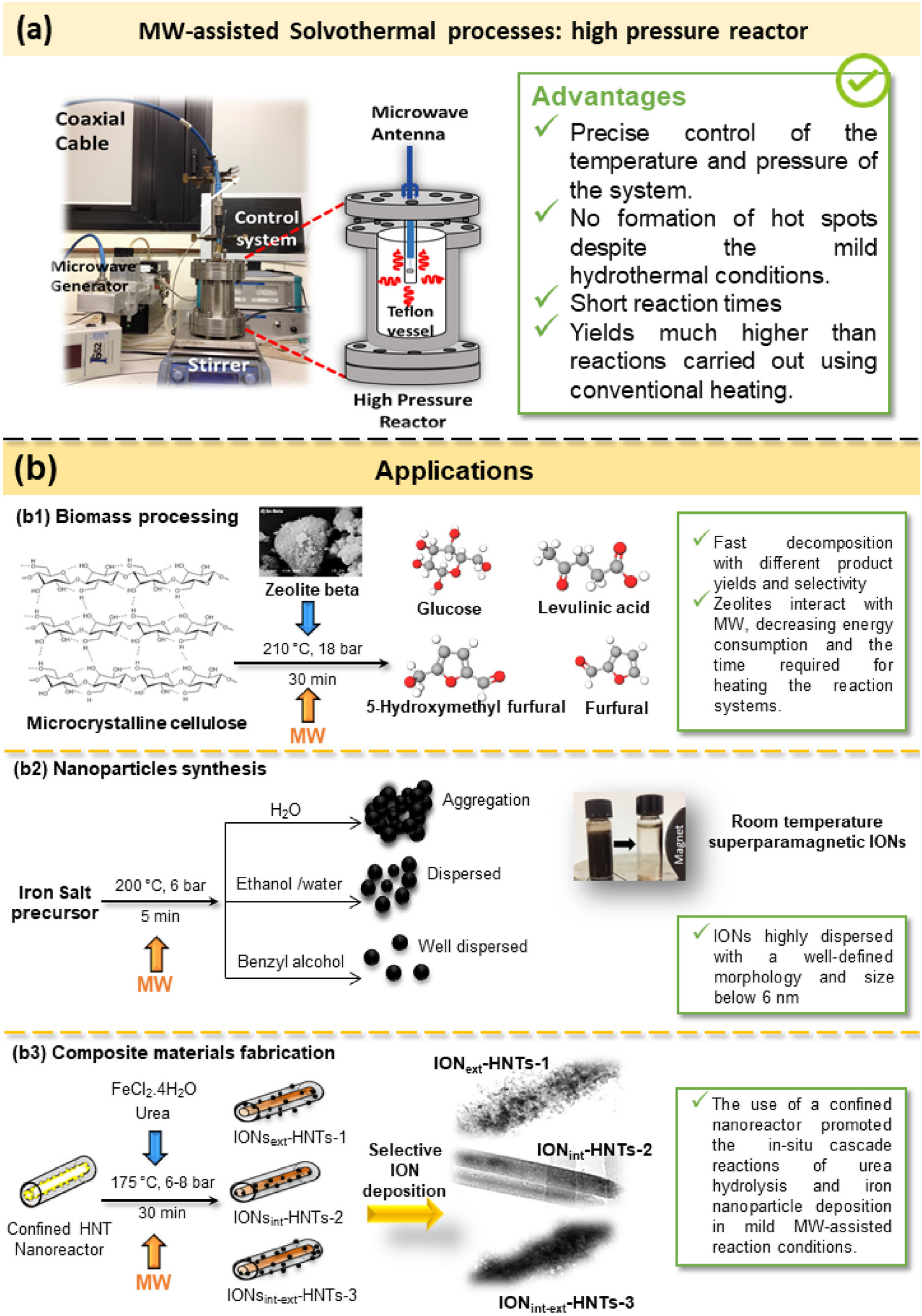

Fig. 4. (a) MW-assisted high-pressure reactor for solvothermal processes. (b) Application of MW-assisted high-pressure to: (b1) hydrothermal heterogeneous catalytic decomposition of microcrystalline cellulose using zeolite beta (b2) Solvothermal syntheses of iron oxide nanoparticles, (b3) Solvothermal syntheses of composite materials.

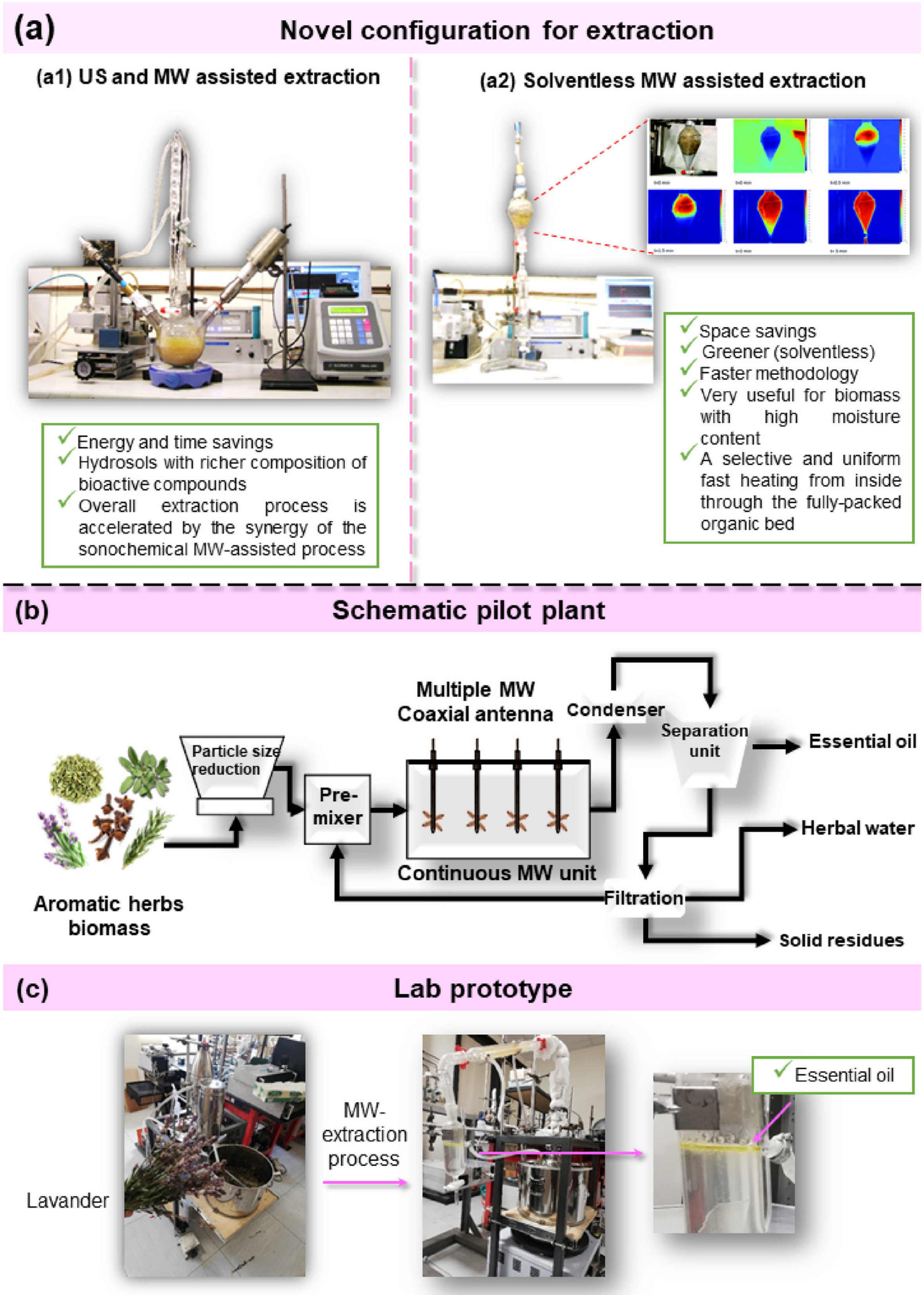

Fig. 5. (a) Novel configuration for the extraction, (a1) US/MW assisted extraction system, (a2) Solventless MW assisted system. (b) Scale up configuration, developed in the framework of Insole project https://in-sole.eu/.

nanoparticles (IONs, Fig. 4-b2) [19], mesoporous materials [17] and magnetothermally responsive composites (built up by halloysite nanotubes and *in situ* IONs deposition, Fig. 4-b3) [20].

5. Novel configurations for MW-assisted extractions

Over the past decade, MW-assisted extraction (MAE) has become particularly attractive mainly because of the fast heating of aqueous samples (due to the strong interaction of water with MWs) promoting low energy consumptions and low cost [21]. Coaxial MW assisted hydrodistillation (MWHD) of EOs from different vegetable materials (leaves of L. nobilis, lavender, rosemary and sage, fennel seeds and clove buds) proved indeed that the method was feasible and cheap. The results showed a significant extraction capability of MW methods for phenylpropanoids and oxygenated monoterpenes greater than the classic MW oven-based approach, different product selectivity, energy saving, and reductions in heating time [2,21].

Usually, US are used to increase the reaction rate of both organic and inorganic synthesis and the yields of extraction processes exploiting the phenomenon of the cavitation. In the simultaneous employment of MW and US the two energetic sources are synergistic [22]. Ragaini et al. developed a new reactor in which MW, delivered by a coaxial dipole antenna, and US, delivered by a metallic horn, can be simultaneously used in for chemical processes in solution (Fig. 5-a1). Using traditional MW ovens or other closed metal waveguides applicator the simultaneous implementation of MW/US irradiation would be difficult or impossible [23].

MW-coaxial antenna technology allows also to conduct solventless MW-assisted extraction on substrates, as biomass wastes, that contain water without the addition of other solvents. Gonzàlez-Rivera et al. developed two new extraction systems for the biorefining of citrus peels: a solventless MW-assisted extraction (SMWAE), and a simultaneous US/MW-assisted hydrodistillation (US-MWHD) apparat (Fig. 5-a2). SMWAE was space saving, clean and fast, while US-MWHD method allowed to obtain a high quality EO [24]. Ascrizzi et al. proposed MWHD and US-MWHD methods and ionic liquids/water mixtures to extract cumin essential oils, developing a method more sustainable, cheaper, and faster. Particularly, the EO composition was comparable, but the yields were higher than those obtained with conventional method [25].

6. Scaling up, frontier applications and future perspectives

The use of coaxial dipole antenna to conduct MW-assisted processes allows to overcome the limitations of the MW ovens and waveguide (i.e., low penetration depth of MW at 2.45 GHz, non-uniform distribution of the electromagnetic fields, formation of hot spots, see Fig. 2-a), and an easier scale-up. The coaxial antennas make possible to irradiate large volumes in batch using non-interfering multiple antennas, placed orthogonally, suitable also for continuous reactors. The electromagnetic field is more uniform than in MW ovens and there is no hot spot formation, thus providing high quality thermal treatments in a variety of reaction vessels (Teflon, quartz, plastic, etc.) [2].

In the framework of Insole project, a first pilot lab prototype (Fig. 5-c) allowed us to obtain EOs from different aromatic herbs and citrus peels with high purity and short times in a full controlled MAE process. Moreover, the development of a pilot plant with multiple coaxial dipole antenna (schematized in Fig. 5-b) is currently in progress.

7. Conclusion

Twenty years of R&D of the high versatile MW coaxial technology has led to the design of different MW-assisted reactor configurations that can be used to perform many different chemical processes, contributing substantially to the development of environmentally friendly methods. The strength points of this MW-technology give the opportunity of designing innovative reactors, their industrial scale-up, and of exploring new applications of MW-assisted processes. The coaxial MW technology is a mature, ecofriendly, cheap, easily implementable alternative to the classical MW oven type reactors.

CRediT authorship contribution statement

José Gonzalez Rivera: Conceptualization, Methodology, Data curation, Writing – original draft. **Elena Pulidori:** Conceptualization, Methodology, Investigation, Data curation, Writing – original draft. **Chiara Pelosi:** Conceptualization, Methodology, Data curation, Writing – original draft. **Carlo Ferrari:** Writing – review & editing, Supervision, Funding acquisition. **Luca Bernazzani:** Writing – review & editing, Supervision, Funding acquisition. **Maria Rosaria Tinè:** Writing – review & editing, Supervision, Funding acquisition. **Emilia Bramanti:** Writing – review & editing, Supervision, Funding acquisition. **Celia Duce:** Writing – review & editing, Supervision, Funding acquisition.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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