AMPERE NEWSLETTER

A Newsletter devoted to RF and MW heating in the range 1MHz to 20 GHz ISSN 1361-8598

www.ampereeurope.org

ISSUE 65
JULY 2010

EDITORIAL

This issue is delighted to welcome two colleagues from France who describe their involvement with RF and Microwaves at their respective bases. Dr Marilena Radoiu who just over a year ago joined SAIREM, describes the scale up of microwave assisted processes. Specifically, she highlights the LABOTRON™ for laboratory scale experimentation and the microwave-assisted 915 MHz installation both for extraction and synthesis with Batch & Continuous flow reactors.

In a similar vain Dr Jean Francois Rochas describes their long involvement at CETIAT in facilitating the technology transfer of industrial processes utilising RF and microwaves. Two projects are highlighted, that of microwave baking of crustless sandwich loaf and a HF vapour pressure timber-drying kiln.

Both colleagues are members of AMPERE's management Committee elected at the OGM in Karlsruhe.

Ricky Metaxas
Editor
EUG St John's College
Cambridge UK

THE SCALE-UP OF MICROWAVE-ASSISTED PROCESSES

by Dr Marilena Radoiu, Sairem, France

The use of microwave energy as a tool for extraction and synthesis chemistry has received increasing attention during the last 10 years or so; nowadays, more and more researchers are applying the microwave technology to a wide range of reactions. To scale-up the microwave technology for unit processes, quantitative characterization has to be successfully carried out for: (a) electric field profile in the microwave reactor, and (b) the resulting changes occurring in the substrate. However, these are complex tasks, difficult to achieve in industrial microwave equipments due to their poor reproducibility and thus giving rise to poor control vs. process quality.

Summarizing from the current literature, the complexity in scaling-up microwave processes arises from the following:

- The theoretical modeling of an empty microwave cavity has little predicting capability for events in a partially loaded cavity due to enormous perturbation of electric field by the very presence of a load;
- The dielectric constant of a given compound varies spatially depending on the chemical composition and bulk density of the load as well as temperature variation during the process;
- Low penetration depth of microwaves giving rise to competing heat transfer mechanisms. Uniform heating is rarely achievable in conventional microwave systems, often giving rise to both unprocessed

and severely over heated spots;
- Various mixing operations applied in mainstream thermal technologies are inapplicable in microwave systems due to material selection issues imposed by microwave environment.

In addition to the microwave complexity, there are other 'more conventional' aspects to be considered when designing industrial chemical equipment for microwave-assisted processing such as: safety, corrosion, up time, maintenance intervals etc.

Based on previous successful microwave-assisted industrial applications, e.g. Laurydoneâ synthesis [1], nitroglycerin removal from the waste acid resulted from nitrocellulose fabrication [2], our current R&D attempts to overcome all the scale-up problems in a simple and reproducible design towards better process quality control at industrial scale:
a) The design of a microwave reactor that overcomes the non-homogeneity profile of the electric field and the non-uniform heating of materials caused by the shallow penetration of microwaves, with a high level of control especially of the temperature inside the reaction mixture to avoid overheating (non-homogeneity may lead to hot spots and degradation of products and the reactor – usually made out of PTFE);
b) The design of a microwave reactor that is easily adaptable to different chemistries: batch, flow, elevated temperature and pressure, safe to use and with a high degree of flexibility;
c) The possibility to run reactions automatically in a continuous and repetitive manner;
d) The design of experimental methods to follow the process after different periods of microwave exposure by characterizing the products and making a comparative study with the existent published data.

The LABOTRON - SAIREM's solution at 2.45 GHz

The LABOTRONTM (Fig. 1) is a system designed to perform microwave-assisted synthesis and extraction. There are two important features of this equipment:

a) The microwave energy is transmitted in to the reaction via an internal transmission line (INTLI) [3]. This method allows for the microwave energy to be transmitted directly in to the reaction using high density electromagnetic fields adapted to each reaction mixture and as such, the availability of very high densities of activation energy. The new modality for microwave transmission also makes it easy to build reactors out of metals (e.g. stainless steel, hastelloy etc.) which will help with pressure containment but also faster thermal transfer;
b) The U-shaped waveguide [4] that allows the mounting of several types of reactors, standard or custom made, and for a smaller foot-print of the equipment.

Through its features, the LABOTRONTM can be used for a wide variety of substances and have adjustable microwave power output with reflected power reading and automatic tuning as well as temperature control. The LABOTRONTM offers a high level of flexibility and control providing optimized microwave energy efficiency and enhanced safety:

- The LABOTRONTM can equally

function with a batch reactor (volume 1.5 L to 17 L) or continuous flow reactor (from a few mL/min up to several L/hour), the recognition of the reactor type is done via the Labotron's PLC;
- Adjustable power from a few watts to 6 kW;
- Optimized geometry of the INTLI to achieve high power densities inside the reactor;
- Direct reading of forward and reflected power values and calculation of the energy absorbed by the irradiated sample;
- Automatic impedance matching for minimal levels of reflected power;
- Efficient external cooling via a cooling jacket;
- PLC-based controller and touch screen user interface. All system functions and status, including recipe changes, alarms and chemical levels are accessible from the touch screen display;
- Quick-connections for increased flexibility and rapid cleaning & maintenance;
- On-line reagents addition and products removal & sampling;
- Based on a mobile platform for quick laboratory installation and positioning.

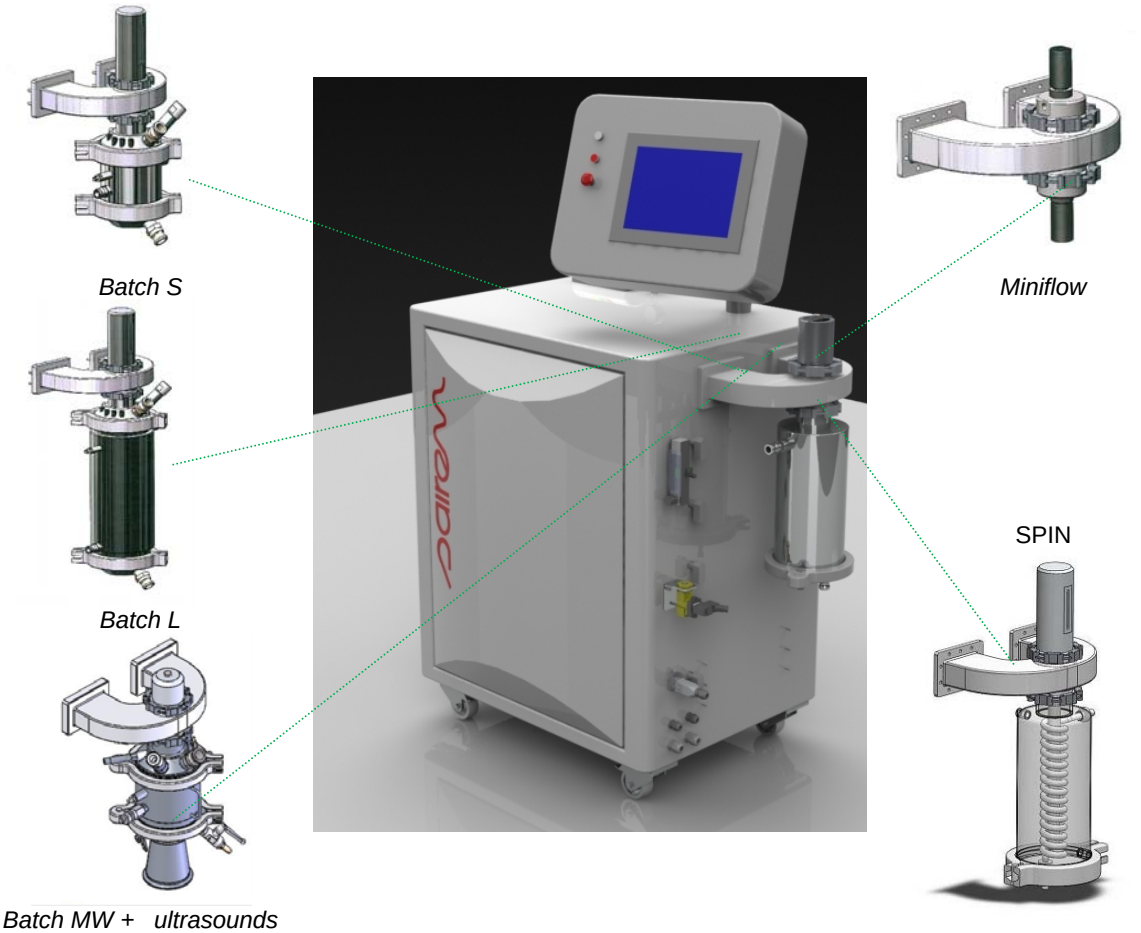


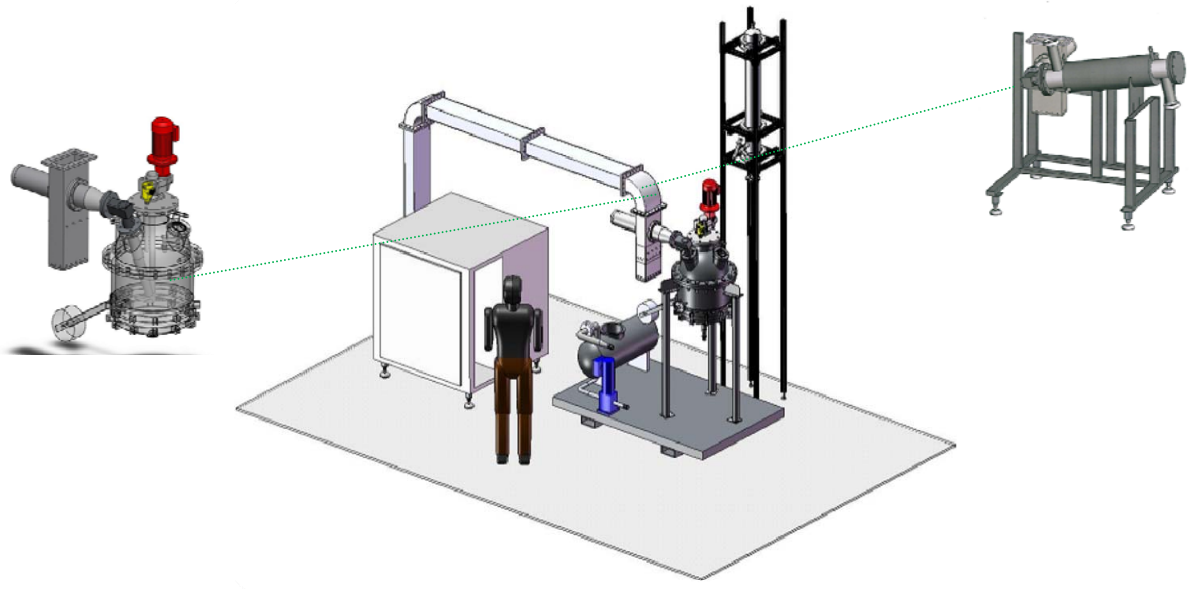
FIGURE 1. *LABOTRONTM 2 & 6 kW EXTRACTION/ SYNTHESIS*

Pilot Instalation at 915 MHz

In collaboration with École Nationale de Chimie in Montpellier, SAIREM have installed a platform unique in Europe for experimentation at a semi-industrial scale.

The main parameters of the pilot-scale installation are:

- Installed maximum microwave power of 30 kW, 915 MHz;
- Fast control of forward and reflected power and high attainable microwave power densities;
- Integration between microwave generator and reactor ensures internal compatibility and control of all system components;
- Continuous flow reactor up to 5 L/min;
- Batch reactor integral with INTLI, with variable speed mechanical stirring, with maximum volume of 100 L adapted particularly for vegetal-type extraction in aqueous phase or solvents;
- Possible recirculation of the extracted product back in to the reaction mixture (loop operation);
- Process could be carried out under an inert atmosphere (N_2, Ar, CO_2 etc.);
- Possibility of on-line filtration or distillation of the products;
- Reactor external cooling via a cooling jacket with automated temperature control;
- *In-situ* temperature measurement.



Batch reactor

Continuous flow reactor

FIGURE 2. Microwave-assited 915 MHz installation for extraction and synthesis with Batch & Continuous flow reactors

REFERENCES
[1] BioEurope SA Groupe Solabia (founiseur pour L'Oreal), France
[2] PB Clarmont, Belgium
[3] Grange, A, Jacomino, J.M. (2009), Device for electromagnetic radiation treatment of a reactive medium, WO 2009/122101
[4] Grange, A, Jacomino, J.M. (2009), Device for applying electromagnetic energy to a reactive medium, WO 2009/122102

CETIAT: THE PARTNER OF HEATING, VENTILLATION AND AIR CONDITIONING INDUSTRIES

Dr Jean Francois Rochas
Cetiat, France

Founded in 1960, CETIAT is a study, test, calibration and training centre specialised in aeraulics, thermics and acoustics. Its workforce of 136 employees is mainly composed of 55 engineers and managers and 57 technicians. In 2009, its turnover amounted to 12.4 million euro. For its 350 industrial members, CETIAT allocates every year a budget of 2.5 million euro to general interest studies for this profession.

CETIAT is aware of the energetic interest of radiant energies in all industrial sectors and for that reason it implemented in 1985 a technological research structure to gather Infrared (gas and electrical), Radiofrequency (13.56MHz – 27.12MHz) and Microwave (915 MHz – 2450 MHZ) competences and test equipment, as well as measuring equipment associated to these different frequency ranges.

CETIAT's ACTIVITY IN THE RADIANT ENERGY SECTOR

This laboratory (unique in France) provides the indispensable interface for industrial development between research organisations (Universities, CNRS , industrial research centres) and equipment manufacturers of the industrial sectors concerned. Today, in these days of inexorable increase in the energy costs, this structure integrates aeraulics and thermics competences – CETIAT's core activity – to optimise synergy between conventional heating technologies and dielectric heating technologies.
This laboratory is equipped with power test equipments for ISM frequency ranges, measuring equipments (VNA, thermometry, etc.) and electromagnetic & thermal simulation soft (HFSS, FLUENT); it allows industrials to carry out tests on a semi industrial scale.
CETIAT is fully independent from equipment manufacturers and, in a first mode of action, it provides possible users asking for it with the means and expertise that will allow them to validate the technical and economical interest of these processes. Once studies are complete, the requesting industrial has in its possession all the items of a specification that can then be distributed to RF or microwave equipment manufacturers in

the form of a call for tender. In this phase, CETIAT acts as a consultant with its customers.
The second mode of action consists in carrying out these studies on behalf of RF or microwave equipment manufacturers or of equipment manufacturers from other branches who want to integrate these technologies into their facilities. For the latter, in addition to the wave/material behaviour study and the implementation of feasibility tests, CETIAT's contribution can even consist in assistance in the design of electromagnetic and thermal parts of machines.
As from its creation, CETIAT's radiant energy application platform has been contributing to the industrial development of the RF and Microwave technologies through approximately one thousand private contracts in miscellaneous industrial branches such as: chemistry, agri-food, plastic & composite materials, textile, wood & wood derivatives. Among the recent industrial developments which do not fall within the scope of confidentiality agreements and to which CETIAT brought its competences, let us mention:

- ¼ scale prototype of a Vapour pressure timber-drying kiln (Drywood-Sairem Company)
Technology: HF 50 Ohms – 13.56 MHz – Installed power output: 50KW.
- Assistance in the design and development of a crustless sandwich loaf baking oven (Concept Convergence Company)

Microwave technology: 2,450MHz – Installed power output: 256 KW – Production capacity: 2 tonnes/hour.

Microwave baking of crustless sandwich loaf

HF vapour pressure timber-drying kiln

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PIERS 2010

27th Progress in Electromagnetics Research Symposium (PIERS)
5-8 July, 2010 Cambridge USA.
For more information on this and other PIERS venues visit:
http://piers.mit.edu/piers/

IMPI Symposium

44rth Annual Microwave Power Symposium
14-16 July, 2010
Curtis Hotel, Denver Colorado, USA
Phone: +1 (804) 559-6667
Fax: +1 (804) 559-4087
info@impi.org

IDS 2010

17th International Drying Symposium, 3-6 October 2010, Magdeburg, Germany.
Deadline for abstract submission: January 18, 2010

For more details contact:
www.ids2010.de or
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Microwave/flow chemistry conference 2011

Sharm el Sheikh, Egypt, Feb 25-28, 2011.
C. Oliver Kappe
Chairman
Zing Conference on Microwave and Flow Chemistry 2011
Sharm el Sheikh, Egypt, February 25-28, 2011
www.maos.net

ICMAT 2001

Materials Research Society of Singapore will be organizing International Conference on Materials for Advanced Technologies (ICMAT) 2011 from 26 June to 1 July 2011. There will be one of the symposiums on "Microwaves in Science and Engineering Applications" chaired by Profs Dinesh Agrawal and Manoj Gupta. For details please contact Mr Kenneth Tan (icmat@dawnyx.com) or visit
http://www.mrs.org.sg/icmat2011/s37.htm

13th International AMPERE Conference 2011

Microwave and High Frequency Heating, September 2011
Toulouse, ENSEEIHT, France

The Management Committee of AMPERE is pleased to announce that the13th International AMPERE conference on Microwave and High Frequency Heating will be staged in Toulouse during Monday to Thursday September 2011. The conference will be held at the Ecole National Superieure d'Electrotechnique, d'Electronique, d'Informatique, d'Hydraulique, et des Telecommunications

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GCMEA 2012 MAJIC 2st
Global Congress on Microwave Energy Applications
Long Beach California USA
Organised by Microwave Working Group International Committee

Chairperson B Krieger Cober
Electronics USA
http://wwwjemea.org./majic2012/

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Association of Microwave Power in Europe for Research and Education (AMPERE Europe)