Book of Abstracts

3rd Global Congress on Microwave Energy Applications (3GCMEA)

Cartagena (Spain), 25-29 July 2016

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Chair of the 3rd Global Congress on Microwave Energy Applications

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TOPIC 02: DIELECTRIC AND MAGNETIC MEASUREMENTS

TOPIC 03: ENERGY AND ENVIRONMENT

TOPIC 04: ENHANCED CHEMICAL REACTIONS

TOPIC 05: EQUIPMENTS

TOPIC 06: INDUSTRIAL APPLICATIONS, MANUFACTURING, PROCESSING CONTROLS

TOPIC 07: MATERIAL SYNTHESIS

TOPIC 08: MICROWAVE ASSISTED EXTRACTION

TOPIC 09: MICROWAVES IN EVERYDAY LIFE

TOPIC 10: MODELING AND NUMERICAL TECHNIQUES

TOPIC 11: NANOTECHNOLOGY

TOPIC 12: PROCESS CONTROL

TOPIC 13: RADIATION SAFETY

Topic 01: DEVICES

Frequency control over energy efficiency and temperature patterns in solid-state-fed microwave cavities

29.07.2016 09:00 Main Room

Vadim V. Yakolev
Worcester Polytechnic Institute, United States

Design and optimization of an antenna for microwave heating of agglomerates

28.07.2016 11:15 Session A (Room 302)

Takahiro Kinoshita, Atsushi Sugihashi
Nippon Steel & Sumitomo Metal Corporation, 20-1 Shintomi, Futtsu, Chiba Pref., 293-8511 Japan.

In iron making process, microwave can be used as an auxiliary heat source in the hot air furnace for heating of the iron oxide and carbon agglomerates layer [A] during transport by the metal conveyer. The penetration depth of the agglomerates for microwave at 2.45 GHz is about 50 mm and it is much smaller than the thickness of their layer (about 250 mm) since the agglomerates efficiently absorb the microwave. In order to heat the bottom part of the agglomerates layer, the waveguide should be inserted into them. The electromagnetic field is, however, affected by the metal conveyer which is below the agglomerates layer. Therefore, we examined the structure of a slot antenna to give microwave energy efficiently to the agglomerates layer, by means of numerical analysis and experiments under the metal conveyer existence.

microwave heating; slot antenna; modelling; iron making process

[A] H. Oda, et al., Dust Recycling System by the Rotary Hearth Furnace, Nippon Steel Tech. Rep., No. 94, pp. 147, 2006.

Compact microwave cavity for heating of liquid chemical samples at 915 MHz

28.07.2016 11:30 Session A (Room 302)

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Institute of Electronic and Mechanical Engineering, Yuri Gagarin State Technical University of Saratov, 410054 Saratov, 77 Politekhnicheskaya Str., Russia

Electromagnetic energy is a widely s

pread tool of scientific research nowadays. Different microwave applicators with ISM frequency 2.45 GHz are used often for experimental studies in physical chemistry, microbiology, material science, electromagnetic compatibility and etc. Single-mode applicators are intended usually for microwave exposition of only one sample to provide uniformity of heat release in lossy dielectric. Multi-mode resonator cavities are employed

for heating several samples at the same time. The problem of inhomogeneous heating is solved by rotating of samples inside cavity in such multi-mode systems.

Design of a single-mode cavity for microwave irradiation several samples of chemical substances is considered in present study. Modified re-entrant cavity resonator is proposed as a basic unit of microwave heating system. It allows decreasing the sizes of the cavity at operating frequency 915 MHz and increasing uniformity of heat release in the samples. Resonator dominant mode is excited with the help of the coaxial probe.

The return loss, electromagnetic and thermal fields were studied by using 3D numerical models and corresponding commercial software for three liquid substances: water, protein and $C_4H_{10}N$

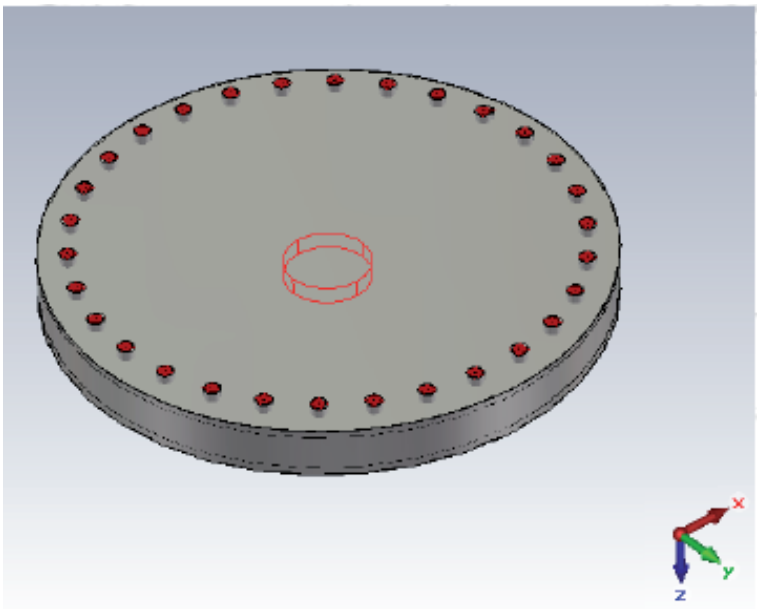
single-mode cavity; microwave heating

Microwave heating pattern beamforming using cylindrical phased-array antennas

ORAL 28.07.2016 11:45 Session A (Room 302)

José Luis Gómez-Tornero, Adriana Pérez-García, Juan Monzó-Cabrera
Dept. Tecnologías de la Información y las Comunicaciones, Universidad Politécnica de Cartagena, Plaza del Hospital 1, 30202 Cartagena, Spain

We present a leaky-wave antenna device inside a cylindrical metallic cavity, which is able to synthesize prescribed near-field microwave patterns for heating/dosimetry applications [A]. This way, a versatile microwave oven can be conceived so that prearranged heating patterns on a given sample can be obtained. A practical example of a 65 cm-diameter 6cm-thick cylindrical microwave oven operating at 2.45 GHz to heat a wet-clay 10 cm-diameter 2 cm-thick sample will be presented. An optimized synthesis procedure will be described to design the slotted-waveguide leaky-wave antenna [B][C][D], which is in charge of generating the desired microwave pattern. CST simulations and leaky-mode theoretical [E] results will be used to demonstrate this interesting device for microwave heating applications.



▲ CST model of cylindrical microwave oven with centered wet-clay sample, excited by leaky-wave antenna model.

microwave heating and dosimetry, leaky-wave antennas, near-field focusing, beam forming

[A] J. Monzó-Cabrera, et al., Kinetics of combined microwave and hot air drying of leather, J. Soc. Leather Technol. Chemists, vol. 84, no. 1, pp. 38-44, 2000.
[B] J.L. Gómez , A. de la Torre, D. Cañete, M. Gugliemi and A.A. Melcón, "Design of Tapered Leaky-Wave Antennas in Hybrid Waveguide-Planar Technology for Millimeter Waveband Applications", IEEE Trans. Antennas Propagat.: Special Issue on Antennas Propag. Applicat. pt.I, Vol.53, No.8, pp.2563-2577, August 2005.
[C] A.J. Martínez-Ros, J.L. Gómez-Tornero, F.J. Clemente-Fernández and J. Monzó-Cabrera, "Microwave Near-Field Focusing Properties of Width-Tapered Microstrip Leaky-Wave Antenna", IEEE Transactions on Antennas and Propagation, vol.61, no.6, pp.2981-2989, June 2013.
[D] J.L. Gómez-Tornero, A.J. Martínez-Ros, and J. Monzó-Cabrera, "Simple Electronic Near-Field Beamforming Using Multi-Tone Microwave Signals with a Leaky-Wave Focused Applicator", IEEE Antennas and Wireless Propagation Letters, vol.14, pp.143-146, February 2015.
[E] J.L.Gómez Tornero, "Analysis and Design of Conformal Tapered Leaky Wave Antennas," IEEE Antennas and Wireless Propagation Letters, vol.10, pp. 1068 - 1071, 2011

Design rules for large scale industrial microwave applicators

ORAL 28.07.2016 12:00 Session A (Room 302)

Guido Link[A], Vasileios Ramopoulos[A], Volker Nuss[A], John Jelonnek[B]
[A] Karlsruhe Institute of Technology (KIT), IHM, Hermann-von-Helmholtz-Platz 1, D-76344 Eggenstein-Leopoldshafen, Germany.
[B] Karlsruhe Institute of Technology (KIT), IHE, Kaiserstraße 12, D-76131 Karlsruhe, Germany.

The benefits of microwave technology with respect to energy efficiency and processing time have been reported for numerous high and low temperature applications, such as drying, cooking, polymers curing or ceramic sintering. Quite often those results base on experiments at lab scale. Upscaling is often challenging, if, even not possible at all. One major obstacle is the need of large scale microwave applicators that allow for homogeneous heating at sufficient heating power.

Large scale applicators are usually resonant microwave cavities. The physical dimensions are typically large compared to the wavelength of the electromagnetic field. Typically, a high number of eigenmodes of the cavity are excited. As a result, a significantly more homogeneous field distribution is available if comparing to smaller applicators. Nevertheless, batch processing in such systems inevitably results in an electromagnetic field with more or less even distribution of standing wave patterns. In combination with a nonlinear thermal response of dielectric absorption in processed materials this may result in fatal local thermal runaway and hot spot formation, and, hence, in significant process limitations. Possible options to overcome this issue are mode stirring devices, movable loads or the intelligent control of microwave sources, distributed along the applicator walls [A]. Otherwise in case numerous microwave antennas and coupling ports should be used, the influence on the overall system efficiency have to be taken into account, in particular for processing of small or low loss loads.

The consequence is that for optimization of large scale microwave systems, parameters like applicator size, installed microwave power and number of antennas always have to be balanced with the permittivity and size of materials and the requested temperature profile for the process. A simple analytical approximation of the system efficiency based on those parameters has been developed and will be discussed and compared to experimental numerical findings with large scale industrial applicators.

microwave applicators; design principles; process efficiency

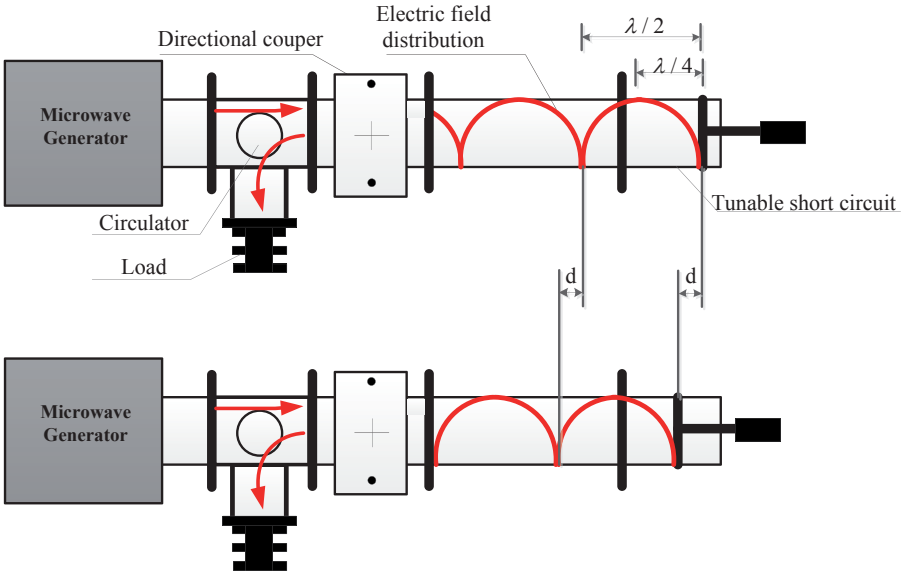
A Sun, Y.; et al.; A novel temperature control approach of distributed microwave feeding systems; Baake, E. [Hrsg.]; Proceedings of the International Scientific Colloquium 'Modelling for Electromagnetic Processing', Hannover, September 16-19, 2014 Hannover: Leibniz University, 2014 pp.155-161.

The effect of moving metal wall on microwave heating uniformity

ORAL 28.07.2016 12:15 Session A (Room 302)

Zhang Chun, Xiao Guang-Nian, Yang Yang, Hong Tao, Zhu Hua-Cheng, Huang Ka-Ma
College of Electronics & Information, Sichuan University, Chengdu, 610065, China

Microwave heating uniformity has been a main drawback of the microwave heating A, many methods were proposal to solve the non-uniformity heating B C D E F G H. In this paper, the phase of electromagnetic field distribution in the cavity is regulated through movements of the metal wall of a rectangular waveguide, which can avoid the inhomogeneous heating caused by the uneven distribution of electromagnetic field in the rectangular waveguide. In order to obtain uniform heating, the metal boundary of the rectangular waveguide was moved at a certain velocity that altered the electric field distribution leading to a more uniform distribution in the material and the waveguide. A Nonlinear Coordinate Transformation method was developed for multiphysics simulation of moving boundary heating, which has a significant effect on solving the moving boundary problem of large industrial microwave cavities. A single mode BJ-22 wave guide heating system is used as the reference in this paper to analyze impacts of short circuit plane position on microwave heating uniformity, the experiment system is shown in Fig.1. The experimental results obtained from Infrared camera which shown in Fig.2 verified that the uniformity of microwave heating can be greatly improved by regulating of short circuit plane position, and the space coordinate transformation method can be used to calculate the moving boundary of the microwave cavity.



◀Fig.1. The effect of short circuit on electric field distribution of waveguide

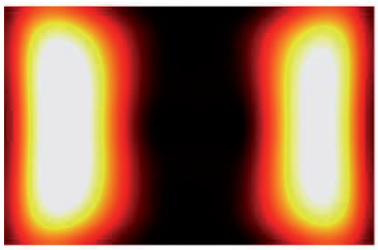
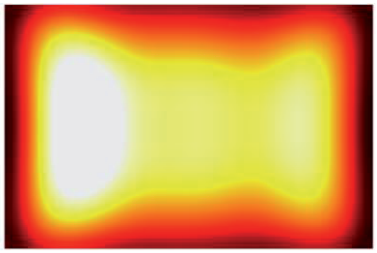


Fig.2. The temperature distribution on the surface after heating 8s.

◀The short circuit without moving

The short circuit with moving▶



microwave heating; heating uniformity; rectangular waveguide; phase adjustment

[A] Guo Q-g, Yang X-q, Yang L-j. Influences of stir and viscosity coefficient on the formation of hotspots during microwave heating, J. Asian Journal of Chemistry, 2010, 22(3):1021-1025.
[B] Pedreño - Molina JL, Monzó - Cabrera J, Catalá - Civera JM. Sample movement optimization for uniform heating in microwave heating ovens, J. International Journal of RF and Microwave Computer - Aided Engineering, 2007, 17(2):142-52.
[C] Chen H, Tang J, Liu F. Simulation model for moving food packages in microwave heating processes using conformal FDTD method, J. Journal of Food Engineering, 2008,88(3):294-305.
[D] Campañone LA, Bava JA, Mascheroni RH. Modeling and process simulation of controlled microwave heating of foods by using of the resonance phenomenon, J. Applied Thermal Engineering, 2014, 73(1):14-23.
[E] Liu CM, Wang QZ, Sakai N. Power and temperature distribution during microwave thawing, simulated by using Maxwell's equations and Lambert's law, J. International journal of food science & technology, 2005, 40(1):9-21.
[F] Yıldız A, Palazoğlu TK, Erdoğdu F. Determination of heat and mass transfer parameters during frying of potato slices, J. Journal of food engineering, 2007,79(1):7-11.
[G] Geedipalli S, Rakesh V, Datta A. Modeling the heating uniformity contributed by a rotating turntable in microwave ovens, J. Journal of Food Engineering, 2007, 82(3):59-68.
[H] Geedipalli S, Datta A, Rakesh V. Heat transfer in a combination microwave–jet impingement oven, J. food and bioproducts processing, 2008,86(1):53-63.

Topic 02: Dielectric and magnetic measurements

New insights about material heating mechanism during microwave processing

26.07.2016 10:00 Main Room

José Manuel Catalá-Civera
Instituto ITACA, Universitat Politècnica de València, Camino de Vera s/n, 46022 Valencia, Spain

A dielectric test-set for monitoring curing processes of thermosetting resins

26.07.2016 11:15 Session A (Room 302)

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[A] Dept. Electrical Engineering, State Semarang University, Building E11, Kampus Sekaran, Gunungpati, Semarang 50229, Indonesia.
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[D] IHE, Karlsruhe Institute of Technology (KIT), Kaiserstr. 12, D-76131 Karlsruhe, Germany.

The fast and energy efficient curing of polymer composites by use of microwave technology gained great interest in current research since those materials face a broad range of applications in packaging, automotive, avionics, and engineering [A]. In order to optimize such microwave assisted processes dielectric properties of the resins need to be known and at the same time the optimum temperature profiles for the curing process need to be determined to achieve a maximum degree of curing.

In this work, a test-set for *in-situ* dielectric measurements during curing based on the waveguide transmission-reflection method at 2.45 GHz has been developed that allows temperature dependent measurements up to 200°C. This test-set provides a fully automatic, computer based data acquisition and temperature-control along any preset temperature profile enabling flexible and reproducible investigations of polymer composites curing. By use of this, time and temperature dependent dielectric properties were measured to investigate the curing behavior of the epoxy resin bisphenol-A-diglycidylether and polyetheramine catalyst. Based on the gained dielectric data a model was developed that describes the dielectric behavior during the polymerization process. This model includes the temperature dependent permittivity of the uncured resin, the catalyst as well as of the fully cured thermoset in combination with appropriated mixing rules and a model for the reaction kinetics. Using both, the dielectric constant and the dielectric loss factor resulted in a more accurate model as compared to models based on dielectric loss factor only [B]. Once the kinetic parameters were obtained by fitting to a single dataset of measured permittivities, this model allows the prediction of the dielectric behavior and curing state along any preset temperature profile. Validation with differential scanning calorimetry (DSC) measurements of investigated samples demonstrate a very good agreement with predicted degree of curing.

dielectric properties; curing process of thermosetting resin; curing model; curing kinetics

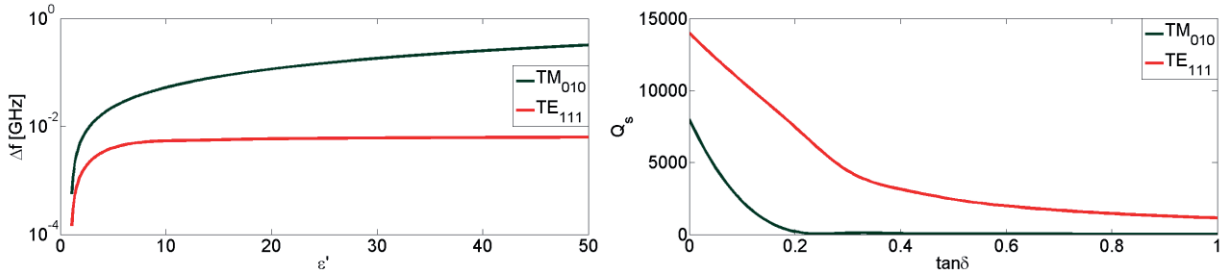
[A] L. E. Feher and M. Thumm. Microwave innovation for industrial composite fabrication—The HEPHAISTOS Technology. IEEE Trans. Plasma Sci., vol. 32, no. 1, pp. 73 - 79, Feb 2004.
[B] R. Hardis, J. L. P. Jessop, F. E. Peters, M. R. Kessler. Cure kinetics characterization and monitoring of an epoxy resin using DSC, Raman spectroscopy, and DEA. Composite, Elsevier, Part A, vol. 49, pp. 100 - 108, 2013.

Advanced dual-mode resonator technique for *in-situ* dielectric measurements of lossy materials

26.07.2016 11:30 Session A (Room 302)

Vasileios Ramopoulos
Karlsruhe Institute of Technology (KIT), Kaiserstr. 12, D - 76131 Karlsruhe, Germany

The successful system design and process setup of a microwave heating system requires the detailed knowledge of the dielectric properties of the processed materials versus temperature. An in-situ measurement of the dielectric properties of the material during heating offers the unique possibility to get the required information on the time-transient behavior of the material properties. It is determined by the simultaneous heating of the material from room temperature up to the maximum processing temperature using high power microwaves together with the measurements of the materials parameters using low power RF measurements. A few measurements systems of such kind based on the cavity perturbation method have been reported during past years already [A][B]. Those are commonly used dielectric materials with low permittivity values only, because of the non-linear and convergent behavior of the frequency shift Δf (or quality factor Q) with increasing dielectric constant (or loss factor), as shown in figure. Different to that, in this paper the simultaneous use of the TM_{010} and TE_{111} modes at 2.45 GHz in a cylindrical cavity with 88 mm in diameter and 88 mm in height and a full 3D simulation of the resonance behavior allows extending the dielectric characterization to significantly higher permittivities. To do so the significantly increased sensitivity of the TM010-mode for measurement of dielectric constant and of the TE_{111}-mode for dielectric loss measurements was exploited, which is going along with frequency shift Δf and quality factor Q, respectively (see figure). The developed measurement setup was successfully tested for high loss materials such as ethylene glycol and water. The measurement accuracy at high permittivities will be discussed and in-situ measurements during the microwave heating process will be presented.



▲ Simulated $\Delta(\varepsilon')$ (left) and $Q(tan\delta)$ (right) for a cylindrical sample with 3 mm in diameter and a length of 90 mm

in-situ; dual mode cavity; dielectric measurements; high permittivity

[A] A. Nesbitt, et al., Development of a microwave calorimeter for simultaneous thermal analysis, infrared spectroscopy and dielectric measurements,IOP Publishing Ltd, pp. 2313-2325, 2004.
[B] J. M. Catala-Civera, et al., Dynamic Measurement of Dielectric Properties of Materials at High Temperature During Microwave Heating in a Dual Mode Cylindrical Cavity, in Microwave Theory and Techniques, IEEE Transactions on , vol.63, no.9, pp.2905-2914, Sept. 2015.

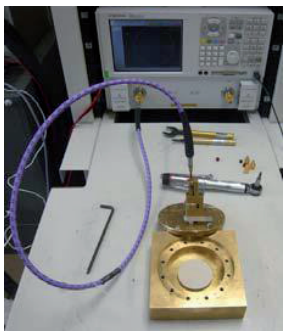
Dielectric measurements with a kind of TM_{0np} at different harmonics

ORAL 26.07.2016 11:45 Session A (Room 302)

Felipe L. Penaranda-Foix, David Marques-Villarroya, Antoni J. Canos-Marin, Jose D. Gutierrez-Cano
Instituto de Aplicaciones de las Tecnologías de la Información y de las Comunicaciones Avanzadas (ITACA), Universidad Politécnica de Valencia, Camino de Vera s/n, 46022, Valencia, Spain.

The measurement of the dielectric constant in materials is the very first thing to do when dealing with microwave technology.

Different techniques are available, and in this paper we present different results of dielectric permittivity measurements done by a cylindrical cavity at multiple harmonics to enlarge the measurement band.



The method to model the cell is based in the circuit theory [A]. The geometry is shown in figure 1 and the circuit model is shown in figure 2. The idea of harmonic values to increase the measurement band was initially introduced by M. Janezic [B].

◀ Fig.1. Circuit Theory applied to TM cavity

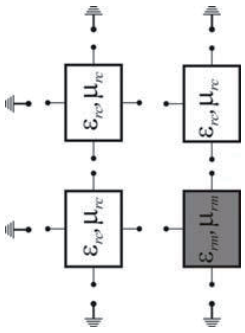


Fig.2. TM cavity ▶

Then the objective is to measure different harmonics of the resonant TM_{0np} modes and then to obtain a wideband permittivity values. Using the facility of the tool designed to estimate the different resonant modes based on the previous information. Figure 3 shows the results for a dielectric sample.

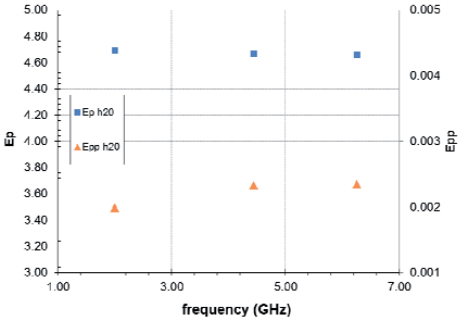


Fig.3. Permittivity at different harmonics

TM_{0np} cavity; dielectric measurements; harmonic resonances

[A] Felipe L. Penaranda - Foix and Jose M. Catala - Civera, "Circuital analysis of cylindrical structures applied to the electromagnetic resolution of resonant cavities", Chapter 7 in Passive Microwave Components and Antennas, 1st ed, Ed. IN - TECH, April 2010 (ISBN 978 - 953 - 307 - 083 - 4). Hardcopy and Online paper are available at webpage: http://sciyo.com/books/show/title/passive - microwave - components - and - antennas.

[B] M. D. Janezic, E. F. Kuester and J. B. Jarvis, "Broadband complex permittivity measurements of dielectric substrates using a split - cylinder resonator". Microwave Symposium Digest, 2004 IEEE MTT - S International; Vol. 3, pp. 1817 - 1820; DOI: 10.1109/MWSYM.2004.1338956. Monzó-Cabrera, et al., Kinetics of combined microwave and hot air drying of leather, J. Soc. Leather Technol. Chemists, vol. 84, no. 1, pp. 38-44, 2000.

Dynamic measurement of dielectric properties of different zeolite Y at high temperature

ORAL 26.07.2016 12:00 Session A (Room 302)

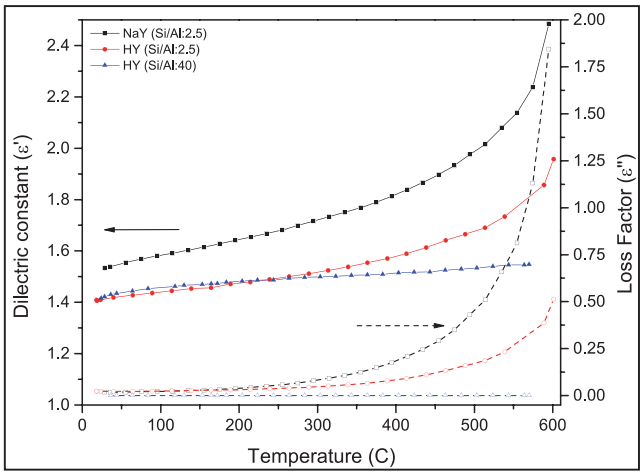
Hakan Nigar[A], Beatriz Garcia-Baños[B], Felipe L. Peñaranda-Foix[B], Jose M. Catalá-Civera[B], Reyes Mallada[A], Jesus Santamaría[A]

[A] Dept. of Chemical and Environmental Engineering and Nanoscience Institute of Aragón, University of Zaragoza, 50018 Zaragoza, Spain

[B] Instituto ITACA, Universitat Politècnica de València, Camino de Vera s/n, 46022 Valencia, Spain

The use of microwaves for the sorbent regeneration is already established as an alternative way of heating solids efficiently. Since microwave heating occurs in a volumetric manner, dielectric materials could be quickly and internally heated. This is the most important step regarding the economic and technical viability of the process [A][B]. Dielectric properties of the material must be obtained for the optimization of the microwave process. Zeolite materials are well known materials as sorbents, catalysts and catalytic supports. Their dielectric properties depend on their water uptake, structure and cation mobility [C]. In this study, three different commercial Zeolite Y powders with different Si/Al ratios and compensating cations were investigated. Dielectric properties (ε' and ε'') of zeolites were obtained using a dual-mode cylindrical cavity, where heating and testing were

performed by two different swept frequency microwave sources. Then, dielectric properties of materials as a function of temperature were calculated by an improved cavity perturbation method during the microwave heating [D]. Figure shows the dielectric properties of the different zeolite samples. HY (Si/Al:40) zeolite showed stable characteristics during the Microwave heating while thermal runaway was occurred in the NaY (Si/Al:2.5) zeolite.



▲ Dielectric properties Vs. temperature

dielectric properties; microwave heating; zeolites

[A] Nigar, H., et al., Removal of VOCs at trace concentration levels from humid air by Microwave Swing Adsorption, kinetics and proper sorbent selection. Separation and Purification Technology, 2015. 151: p. 193-200.
[B] Nigar, H., et al., Amine-functionalized mesoporous silica: A material capable of CO_2 adsorption and fast regeneration by microwave heating. AIChE Journal, 2016. 62(2): p. 547-555.
[C] Gracia, J., et al., Heating of zeolites under microwave irradiation: A density functional theory approach to the ion movements responsible of the dielectric loss in Na, K, and Ca A-zeolites. Journal of Physical Chemistry C, 2013. 117(30): p. 15659-15666.
[D] Catala-Civera, J.M., et al., Dynamic Measurement of Dielectric Properties of Materials at High Temperature During Microwave Heating in a Dual Mode Cylindrical Cavity. Microwave Theory and Techniques, IEEE Transactions on, 2015. 63(9): p. 2905-2914.

Wideband permittivity measurement using coaxial line and capacitive cell

ORAL 26.07.2016 12:15 Session A (Room 302)

M. W. Ben Ayoub [AB], E. Georgin [A], J. F. Rochas [A], S. Hubert [A], P. Achard [A], L. Neves [B], P. Sabouroux [B]
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The measurement of the complex dielectric properties of materials at radio-frequencies and in the microwave spectrum is nowadays a major focus in materials metrology, since it allows determining the electromagnetic characteristics of materials (permittivity and/or permeability). These two characteristics are useful in many sectors, such as medical applications like tumor ablation and microwave industrial heating. In addition, these parameters can be used as an intermediate variable to measure another characteristic in product; in our project we use it to reach the moisture content in a solid. This project is part of a European metrology research project called SIB64 METefnet

(http://www.metef.net/) and funded by the European Metrology Research Program Committee (EMRP).

To achieve this goal, we have developed two cells for measuring the dielectric permittivity, non-resonant, non-destructive and which can be used for solids and liquids. The first cell is a capacitive cell which can be used for RF frequencies from 25 kHz to 150 MHz. The effectiveness of the cell was validated with several types of liquids, such as alcohols, and also with a few solid references; these first results are published in [A]. The second is a coaxial cell, used to measure the complex electromagnetic parameters for higher frequencies, in the microwave spectrum. It allows characterizing the material between 50 MHz up to several GHz. The geometrical dimensions of this cell are: inner diameter is equal to 16.4 mm and the outer diameter is equal to 38.8 mm (1"5/8 coaxial line).

This work mainly focus on the coaxial cell. The measurement principle is based on 4 measurements, with the same cell, under different configurations: transmission, matched load, short circuit and open circuit. Then they are used to solve a system of 8 equations leading to ε' and ε'' determination. Also, the Nicolson-Ross [B] and Weir [C] (NRW) method can be used to measure permittivity and permeability with application of a de-embedding process [D], this method only requires the measurement in transmission and in reflection, but it is known that this technique can diverged for low -loss materials. Hence our idea of adding two additional measurements by using a short circuit piston sliding and then choose the most accurate according to the characteristic of the material.

In order to validate the method of measurement, we have the EpsiMu® tool (http://www.epsimu.fr/) which has been developed by the Institut Fresnel. Since it allows characterizing all types of materials in the band [50MHz - 10 GHz], it provides an excellent comparison method for an accurate development of our coaxial cell. So far in the project, the calibration of the cell and first results on solid materials measurement (PTFE, PVC...) has shown the effectiveness of the cell. The current work is about the optimization of the measurement method and testing on different types of products for greater coherence between the measurements obtained with the two cells.

Permittivity; permeability; coaxial cell; microwave measurement

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Permittivity of fresh vegetables smoothies at radiofrequency and microwave frequencies and various temperatures

ORAL 26.07.2016 16:00 Session A (Room 302)

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In this work we have measured the permittivity [A] of different fresh vegetables smoothies at different frequencies and temperatures in the range [20, 80]℃. To measure the dielectric properties we have used the dielectric probe DAK of the SPEAG company in the frequency range [10MHz, 20GHz]. This range covers radiofrequency and microwave frequencies. Obtained results have been compared to those obtained by a dielectric dielectrometer at frequencies close to 2.45 GHz to verify the measurements obtained using the dielectric probe.

Once the measurements have been carried out, Debye and Cole-Cole models of permittivity have been obtained for fresh vegetables smoothies products using an inverse technique to fit the models to the measured values. Debye and Cole-Cole Parameters jointly with conductivity that fit the measured curves are provided and may be useful to model these materials easily.

The authors are grateful to Spanish Ministry of Economy and Competitiveness (MINECO) Project AGL2013-48830-C2-1-R and FEDER for financial support.

microwave; permittivity; radiofrequency; smoothie

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The study on the permittivity of pyridine-ethanol mixed solutions

ORAL 26.07.2016 16:15 Session A (Room 302)

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The experimental measurements of the effective permittivity of the pyridine-ethanol mixed solution has been measured over the whole range of concentrations at 20, 30 and 40℃, respectively. Surprisingly, it is found an exceptional phenomenon that the real part ε_r' and the imaginary part ε_r'' of the mixed solution permittivity are larger than those of their pure components at certain concentration. At the same time, the real part ε_r' of pure pyridine and pure ethanol solution permittivity has been measured at 20℃ covering a frequencies range from 0.2 GHz to 10 GHz. Moreover, for the unusual behaviors of the real part ε_r' and the imaginary part ε_r'' of the mixed solution permittivity, a simple analysis has been given from the angle of the molecular clusters.

complex permittivity; ethanol; pyridine; intermolecular interaction

Microwave heating of powders at high temperature: effect of grain size and bulk density

POSTER 26.07.2016 14:15 e-Poster Session (Show Room)

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The dielectric properties of a material depend mainly on moisture content, temperature, chemical composition and frequency of the applied electromagnetic field. However, factors like bulk density and particle size must also be considered, especially when dealing with powder or granular materials, which are used in many industries, such as the food and agricultural, chemical, mining, construction and pharmaceutical. Knowing the dielectric properties of such materials at different bulk densities would provide useful information for their microwave-assisted processing, since some materials may be processed at different bulk densities for different applications [A].

There are many studies providing mixture equations which relate the dielectric properties to those of the solid material or to the mixture at a volume concentration different from the one tested [A][B]. However, a major problem when using powdered materials is that their dielectric properties not only change at different densities (level of compaction), but also depend on the particle size. Thus one of the main objectives of this study is to determine the joint influence of both parameters (density and particle size) on the dielectric properties at room temperature.

On the other side, it is still unexplored how these two parameters affect the thermal behavior in microwave heating processes. Microwave equipment recently developed [C] allows dielectric characterization of powder materials during microwave heating up to high temperatures (>1000℃). In previous works, the effect of particle size on the temperature curves of microwave-heated samples was evaluated. In this work, the effect of bulk density on the microwave heating is measured and discussed. The dielectric properties of powder samples determined at a wide range of temperatures are also provided.

grain size; bulk density; powder; microwave heating; dielectric properties

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Study of microwave thermal processes through *in-situ* Raman and dielectric spectroscopy

POSTER 26.07.2016 14:15 e-Poster Session (Show Room)

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The use of microwave energy for materials processing is well known, however, the heating mechanisms and the particular thermal behavior of materials under high frequency electromagnetic fields is still a matter of research. Recent works [A] highlight the need of further investigations to understand the microwave-matter interactions behind the materials thermal processes.

Dielectric spectroscopy is a valuable tool to characterize the materials at microwave frequencies and to know how they are influenced by the presence of electromagnetic fields. Extensive research has been done to measure the dielectric properties of materials by many different techniques [B]. Since these properties are temperature-dependent, they change during the heating process. Recent studies [C] allow dielectric characterization of materials in real time during microwave heating processes, up to high temperatures (>10000℃).

The next step proposed in this work is linking the information given by dielectric properties to the thermal processes that occur in the material. To this end, a conventional technique such as Raman spectroscopy is simultaneously applied to obtain information about the microstructure and the chemo-physical changes in the materials.

Characterization results of several selected materials in a wide range of temperatures are presented and discussed. The in situ combination of both techniques (dielectric and Raman) in real time during microwave processing gives useful and innovative information about the heating mechanisms. This knowledge is crucial in several aspects: to analyze the effects of the microwave field on the reaction pathways; to design and optimize microwave-assisted processes, to predict the behavior of materials leading to repeatable and reliable heating processes, etc.

Dielectric; Raman; high temperature; microwave processing

[A] R. Raman Mishra, A. Kumar Sharma, "Microwave–material interaction phenomena: Heating mechanisms, challenges and opportunities in material processing", Composites: Part A 81 (2016) 78–97.

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Topic 03: Energy and environment

Microwave induced pyrolysis for producing syngas

PLENARY 28.07.2016 09:20 Main Room

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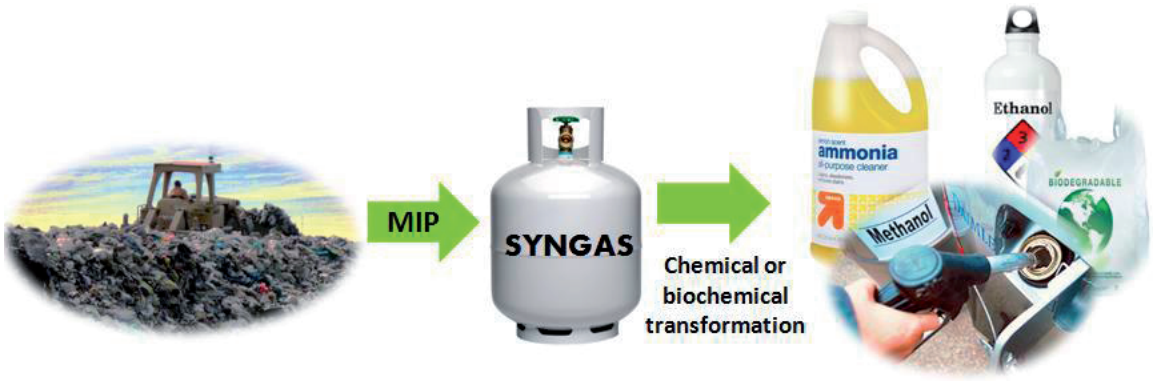
Up to 3000 million tons of waste is generated in the European Union every year. Of these, organic solid wastes have the potential to partially satisfy the production of chemicals, whilst the environmental impact can be minimized and the sustainability of the processes increased compared to those based on fossil resources. However, these wastes are complex due to their high heterogeneity, and new conversion processes are necessary before they can be converted into high value products. One possibility is to process these wastes using the microwave-induced pyrolysis (MIP), as an alternative to the conventional pyrolysis or gasification processes, that leads to the production of simple molecules, mainly H_2 and CO, known as syngas.



This talk will disclose the research carried out at MCAT (INCAR-CSIC) in the last years [A][B][C], which has been aimed to develop a new MIP process that allows conversion of biosolids into syngas. The different organic substrates that can be processed, the operational conditions that lead to a maximum production of syngas and the characteristics of the syngas as well as the by-product obtained will be discussed. A particular emphasis will be put in the partial recycling of the solid fraction and its role as microwave susceptor and catalyst of some of the gasification reactions taking place during the MIP of the organic residues. Additionally, some insights on the energetic costs of the process will also be given.

pyrolysis; biomass; organic residues; syngas; microwave susceptor

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Microwave-induced growth acceleration in plants

ORAL 27.07.2016 11:15 Session A (Room 302)

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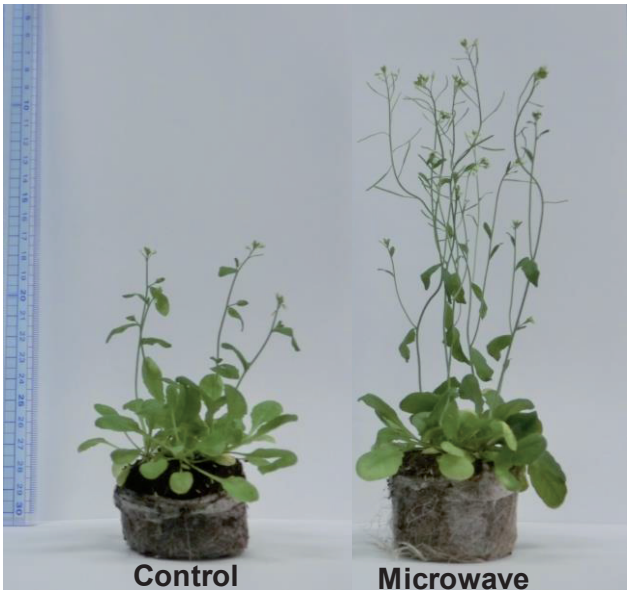
Microwave-induced growth acceleration of plants is reported in this research. The response of plants (Arabidopsis thaliana) to microwave irradiation was investigated to specify the conditions that enhanced plant growth. Microwave irradiation accelerated the phase transition from vegetative to reproductive growth, the growth of the inflorescence stem, as well as seed germination. However, microwave irradiation did not affect plant diameter. In addition, it was found that microwave irradiation enhanced the expression of a gene that regulates growth phase transition, suggesting that microwave irradiation could indeed enhance the growth of plants by modulating the expression of gene. On the other hand, the expression of genes involved in the response of plants to heat stress were not clearly altered by microwave irradiation. Our results suggest that the effects of microwave irradiation on plant growth is not from the thermal effect. By understanding the key factor behind the microwave-dependent growth stimulation, it makes it possible to apply this finding to the growth of other plants (potato, corn, switch glass, etc).

When either microwave heating or conventional heat treatment was applied to A. thaliana, a significant change in the growth of the leaves could not be observed. However, the shift to the reproductive stage and the growth of the inflorescence stem were dramatically promoted in the microwave treatment condition. Because such phenomena were not observed under ordinary high temperature (40℃) conditions, a specific effect that was only observed in the case of microwave irradiation was expected. When the expression of the gene that regulates growth or stress response was investigated, the expression of the gene which controls the growing stage was affected by microwave irradiation. Expression of other certain heat stress response genes, on the other hand, were not altered.

Although the expression of the CO gene was not affected by microwave irradiation, expression of the FT gene was enhanced 4 days following the microwave irradiation. On the other hand, the genes encoding heat shock proteins or MBF1c that are involved in heat

stress responses of plants did not respond to the microwave. From these results we suggest that the effect is not a thermal response of the plant.



◀Grown comparison of Arabidopsis thaliana with no radiation (control) and microwave radiation (Microwave)

Microwave treatment; growth acceleration; arabidopsis thaliana; molecular biology

Microwave pyrolysis of biomass in a fluidised-bed process

ORAL 27.07.2016 11:30 Session A (Room 302)

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Pyrolysis is a thermochemical process during which biomass materials are heated in an inert atmosphere at around 500℃ to produce liquid (bio-oil), solid and gas products [A]. Bio-oil can be used for chemicals and biofuel production. High bio-oil yield requires high heating rates [A]. Microwave heating has been considered as a promising technique for providing the energy required for biomass pyrolysis due to its volumetric and selective heating nature which allows for rapid heating in a cold environment. One of the major challenges facing microwave pyrolysis is its heating heterogeneity which is caused by the distribution of the electric field inside the heating cavity and penetration depth of the resultant solid product [B][C]. To overcome this challenge, a microwave-heated fluidised-bed process was developed to improve heating homogeneity by creating continuous movement of the particles within the applicator.

The biomass material used in this study was sycamore. Cold fluidisation experiments showed that sycamore particles in the 0.6 to 1.18 mm range fluidise following group B behaviour in Geldart classification [D]. Larger particles needed very high gas velocities to fluidise leading to complex and turbulent fluidisation behaviour. Pyrolysis experiments

showed that particles with the same size fluidise at much lower gas velocities due to a reduction in size and density during heating.

The developed process involves heating a fluidised-bed of biomass particles inside a multimode cavity. The process design was based on the knowledge of the dielectric properties of the studied biomass material, its fluidisation behaviour and the pyrolysis reaction requirements. Nitrogen at room temperature (20℃) was used as the fluidising gas. It was found that the energy required to volatilise 60-70% of the solid is around 3.5-4.0 kJ/g. The amount of solid volatilised was found to increase with increasing the particle size and decrease when the gas velocity is increased above the minimum fluidisation velocity.

This study shows that the heating heterogeneity challenge associated with microwave pyrolysis can be overcome through processing in a fluidised-bed system which opens up significant opportunities for further work towards large-scale biomass pyrolysis using microwave heating.

microwave; pyrolysis; biomass; fluidised bed

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Impact of microwave susceptor on biochar properties obtained from biosolids MWAP

ORAL 27.07.2016 11:45 Session A (Room 302)

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Microwave assisted pyrolysis (MWAP) is an environmentally friendly approach to convert biosolids into value-added products, such as biochar, biogas and bio-oil. Biochar is a stable material rich in carbon [A]. Interest in this product has been increasing due to the potential benefits in many different applications: soil ameliorant, contaminant removal and as a microwave absorber. Biochar has been particularly explored as a soil ameliorant to enhance soil physicochemical properties, consequently improving crop productivity, nutrient availability and water holding capacity [B]. Specific surface area (BET) plays a key role in the biochar performance in these applications, particularly in adsorption or diffusion processes.

However, dry biosolids are almost transparent to microwave irradiation, and an addition of a microwave susceptor is needed to achieve the temperature required for pyrolysis. A microwave susceptor is a material able to absorb electromagnetic energy and re-emit this energy as heat to the surroundings; the efficiency of this process is determined by the loss tangent of each material. The main objective of this work was to study the impact of two microwave susceptors (activated carbon and glycerol with loss tangent 1.71 and 0.71 respectively) on microwave pyrolysis process and biochar properties. These two microwave susceptors were mixed in different proportions (10% and 20%) with biosolids and pyrolysed at different temperatures (400℃, 600℃ and 800℃). The heating rate of biosolids mixed with glycerol was significantly lower than activated carbon; however, after 400℃, the heating rate of the two mixtures was similar due to the formation of biochar, which trigger the heating rate because of its good microwave absorption properties. The change in the properties of the biochar can be attributed to the difference in the dielectric properties of the initiator and hence heating rate.

Dielectric properties of biosolids, microwave susceptors and biochar were analyzed. Chemical (FTIR, ICP, XRD) and physical analysis (SEM, BET) were carried out on the final biochar. In general, the BET of biochar produced with glycerol was considerably lower than with activated carbon. The chemical composition and crystalline structures of biochar were similar in both cases; however, FTIR results showed the presence of aliphatic and O-H bonds in biochar produced with glycerol.

Biosolids; biochar; microwave susceptor; pyrolysis

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Microwave-driven plasma gasification for biomass waste treatment

ORAL 27.07.2016 12:00 Session A (Room 302)

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Gasification technology may combine waste treatment with energy generation. Conventional gasification processes are bulky and inflexible. By using an external energy source, in the form of microwave-generated plasma, equipment size may be reduced and flexibility as regards the feed composition may be increased. This type of gasification may be combined with fuel cell technology to generate electricity for on-site microwave generation.

In this work, we present gasification experiments with several biomass feedstocks in an air plasma. In order to optimize reaction rates and minimize equipment size, a plasma generator is integrated into an entrained flow gasification type of arrangement. In this manner the feedstock is exposed to a maximum intensity plasma to enable gasification over very short residence times.

At the present small scale of ~0.3 L processing volume, continuous flow syngas generation experiments have resulted in cold gas efficiencies of up to 40 % at ~90% conversion. Heat and mass transfer phenomena have been characterized and our results have been extrapolated in scale-up studies.

plasma gasification; waste valorization; energy from biomass; microwave plasma

Arrangements for radio-frequency heating of building structures

27.07.2016 12:15 Session A (Room 302)

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Radio-frequency (RF) heating (in our case with 13.56 MHz) is a suitable tool for heating building structures in a controlled manner because homogeneous heating with sufficient penetration depths can be achieved. In contrast to microwave heating (e.g. using 2.45 GHz), a wide spectrum of dry as well as moist materials such as concrete, stone, brickwork, limestone or timber can be treated. This can be applied for thermal enhancement of drying (e.g. after flooding events) or decontamination [A]. Additionally, moderate heating to about 60°C is an eco-friendly alternative to the application of hazardous chemicals for pest control [B].

While RF heating of plane masonry is relatively simple when using parallel plate electrodes with direct contact via an electronic matching network to the generator, special arrangements have to be developed: (i) when the target structures are accessible from only one side (e.g. a parquet floor), (ii) when contacting of the electrodes through the wall via boreholes is not acceptable (e.g. for historical buildings) or (iii) when the structure is confined by a medium such as soil or water on one side and therefore not accessible for conventional electrode installation (e.g. basement ground).

In order to solve these problems, two special electrode designs have been developed by modelling the field distribution and energy absorption and by carrying out test experiments to validate the simulation results. One solution is based on a two-dimensional surface capacitor structure providing certain penetration depths and being especially suitable for

treating thin structures such as wooden parquet floor. This arrangement can be particularly used for pest control. When cooling the surface and realizing heating in the volume, RF heating can then also be used for sensitive sealed surfaces. The other solution uses a capacitive coupling between the grounded shielding and an electrode or an equivalent structure (e.g. moist soil) at the other side of the masonry in order to establish a sufficiently strong electrical field between a "hot" electrode on the side of the shielding and the coupled rear electrode.

Both arrangements have already been successfully tested in the laboratory and the field scale. The obtained temperatures in the volume of the building structures are sufficient to achieve drying or decontamination as well as elimination of pests.

radio-frequency heating; drying; decontamination; pest control

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Microwave-assisted energy-saving: organic hydride system for hydrogen energy storage

27.07.2016 16:00 Session A (Room 302)

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By the year 2050, Japan's major target is to reduce the emission of the greenhouse gas CO_2 by 50%, and the introduction of renewable energy sources is indispensable to achieve this goal. One technology that is highly expected is hydrogen-based fuel cell technology; accordingly, fuel cells have been, and still are one of the targets of extensive studies in Japan. Certain public institutions (e.g. in transportation sectors such as buses) already make use of fuel cells as a source of power. Moreover, private cars that operate using fuel cell technology (fuel cell vehicle: FCV) are already commercially available (Toyota Motor Co. since December 2014). Power supplied by fuel cells is widely used in Japanese homes as well. The hydrogen gas is first extracted from liquefied petroleum gas (LPG), and its energy is subsequently converted by the fuel cell system into electricity (Ene farm system, Panasonic Co.). The processes occurring in a fuel cell are exothermic; the heat used to boil the water could then used in household applications, such as bathrooms. Although it may not be as obvious, hydrogen energy is very abundant our daily activities today.

This presentation will describe the process of producing hydrogen gas from methylcyclohexane (MCH) as organic hydrides using a microwave-assisted selective heating process. This process requires a catalyst, which consists of platinum particles supported on an activated carbon substrate (Pt/AC) [A][B][C][D][E]. When utilizing microwave

radiation for dehydrogenation of MCH (methylcyclohexane), the efficiency of dehydrogenation resulted in over 94%. In this condition, the Pd/AC catalyst bed heated to ca. 340℃ only within ca. 2 min (for 12 W applied MW power). When using conventional heating (ceramics heater) on the other hand, it required ca. 35 min for the catalyst bed to reach the efficient temperature of reaction; this was the case even when the reactor was insulated to suppress the loss of heat. The microwave-assisted process made it possible for hydrogen evolution to occur in much shorter times, saving a considerable amount of electric energy (ca. 75 %): the total power consumed using the microwave system was 33 W (microwave applied power, 12 W), while the ceramics heater expended 139 W of power. From these results the advantage of using microwave heating is clear, as electrical energy needs to be consumed to produce hydrogen energy.

heterogeneous catalyst; dehydrogenation; hydrogen energy; organic hydride

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Reduction of CO_2 with hydrogen in a microwave-driven plasma reactor

ORAL 27.07.2016 16:15 Session A (Room 302)

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Two major environmental and scientific challenges faced nowadays are energy storage and greenhouse gas emissions. Plasma-based technologies have the potential to enable storage of renewable energy by means of converting greenhouse gases into synthetic fuels. A microwave-driven plasma reactor was investigated as an alternative to reduce carbon dioxide (CO2) with hydrogen (H_2) to carbon monoxide (CO) through the reverse-water-gas-shift reaction (RWGS: $CO_2 + H_2 \leftrightarrow CO + H_2O$). A further reduction of CO by the Fischer-Tropsch synthesis (FTS) can lead to the production of hydrocarbons, which can be easily transported and stored. The main objective was to assess the performance of the chemical reaction by tuning input variables (mixture ratio, flow rate, pressure, input microwave

power) and also plasma-related parameters. Indeed, plasma sources can provide both energy and highly reactive species simultaneously, thus enhancing chemical reaction performance [A].

In this work, an electromagnetic surface-wave launcher (Surfatron50, Sairem) in combination with a solid state microwave generator (MiniFlow 200S, Sairem) was used to perform the RWGS reaction in a non-catalytic plasma reactor. High CO_2 conversion rates of about 85% were observed when the feed gas mixture (H2:CO2) was equal to 3. Therefore, the addition of molecules with higher energy content such as H2 improved the dissociation process, thus acting as a physical catalyst. A ratio H_2:CO of around 2 was obtained in the product stream under the same conditions. Moreover, no by-product formation was seen as compared to previous studies [B]. These features make the product gas suitable to directly feed it into FTS. Concerning the energy efficiency, it must be noted that this lab scale system was not designed to achieve high energy efficiencies, as its main purpose was to facilitate plasma ignition. In fact, the maximum energy efficiency (electrical energy into chemical energy) was about 8%. With regard to the selectivity, it was noticed that the selectivity for CO was slightly higher than the selectivity for water. Lastly, it is remarkable that high conversion rates were achieved at milder operating conditions using non-thermal plasma (bulk gas temperature close to room temperature) whereas the conventional process requires about 1000 K to reach the same conversion [C].

electricity storage; CO_2 utilization; microwave plasma reactor; process intensification

[A] Li et al. Carbon dioxide reforming of methane using DC Corona Discharge Plasma Reaction. J. Phys. Chem. A, 108, 1687-1693 (2004).
[B] L. Maya. Plasma-assisted reduction of carbon dioxide in the gas phase. J. Vac. Sci. Technol. A 18, 285 (2000).
[C] M. De Falco et al. Methanol production from CO2 via reverse-water-gas-shift reaction. Green Energy and Technology. (2013).

Microwave treatment of asbestos and other wastes

ORAL 27.07.2016 16:30 Session A (Room 302)

Ryszard Parosa
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Asbestos is a mineral of fibrous structure. Because of its properties, such as being fire and chemical proof, poor thermal conductivity and low price, it was used worldwide in industry, construction, transport and others. It appeared, however, that asbestos fibers – very thin and light – are easy to inhale and can cause serious diseases, e.g. asbestosis and lung cancer. For this reason, asbestos waste is classified as a hazardous group, and materials containing asbestos are being withdrawn from any application. At present, the most widely

used method of disposal of asbestos waste is isolating it from the environment through packaging of dismantled elements in plastic bags, which are then transported and deposited on special landfills.

The MTT (Microwave Thermal Treatment), which is the basis of the described technology, is the thermal destruction of asbestos fibers. Asbestos, heated to temperatures of around 1100℃, loses its fibrous structure because in these conditions fibers are destroyed (remineralisation), and the obtained material loses its harmful properties. However, heating of asbestos to high temperatures requires a relatively high energy, and is expensive as well, because asbestos is a good heat insulator. The essence of MTT method is the use of intensive microwave radiation to heat asbestos waste. Microwaves penetrating deeply into the material heat its entire volume. In that way, a significant reduction in costs is obtained. Chemical substances admixed to waste cause better absorption of microwaves and reduce the temperature of remineralisation to about 900℃, which further improves the process's economics.

The process is performed with the use of two kinds of reactors: HR 200 with efficiency of about 150-200 kg/hour, and HR 5000 with efficiency of about 3 tons/hour. HR 200 reactor is installed inside the standard container and can be transported to the site where asbestos waste has occurred. The system is equipped with 8 microwave generators with output of 3 kW each (2.45 GHz). HR 500 reactor is equipped with one or two microwave generators: 75 kW or 100 kW (915 MHz).

Exhaust gases from HR reactors are cleaned in another reactor called MOS (Microwave Oxidation System), constructed in the form of a cylindrical vessel filled up with ceramic elements absorbing microwaves. Ceramic elements are heated by microwaves up to ca. 1000℃.

The same system has been successfully used for the treatment of other kinds of waste, such as municipal waste, sludge, cleaning of soil polluted with oils, and hospital waste. Through some modification of the system (HR reactor + MOS reactor), several processes have been provided: burning of wastes, gasification or pyrolysis.

microwave application; microwave heating; waste treatment

[A] R. Parosa, Application of MTT metod and MOS for rendering of asbestos waste (Zastosowanie metod MTT I MOS w unieszkodliwianiu odpadów azbestowych), GAJA publication, 2011, Wrocław, Poland.
[B] Y.Yvon, P. Sharrock, J.Y.Cojan. M.C. Magnie, Physico-Chemical treatment of asbestos containg materials, 3rd International Conference on Engineering for Waste and Biomass Valorisation, May 17-19, Beijing, China.

Effectiveness of innovative microwave wood modification technology applications in industry

28.07.2016 14:15 e-Poster Session (Show Room)

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Very low permeability of many wood species cause problems in the wood processing industries. These include: very long processing times, large material losses, high energy consumption, and environment pollution. Microwave wood (MW) modification technology provides an increase in wood permeability for liquids and gases that solves many of these problems.

The research objectives of this study were to quantify the effect of MW wood modification technology on economic aspects of material production. In particular, the aim was to analyse the effect of electricity, labour and equipment costs on MW processing costs at different production scales. Three material production schemes with MW treatment stages were used for economic modeling of new technology applications: pulp production, lumber drying and railway sleeper manufacturing.

Results of economic study showed significant advantages of technology use in pulp and timber industries. MW technology application in different pulp mill conditions can provide a reduction in the softwood chemical pulp production costs by 18.3 – 27.1%. Specific costs of softwood chip processing in MW plant at electricity costs 0.06 – 0.12 US$/kWh are 23.6 -35.2 US$/ADT (air dry ton) of pulp.

The cost of MW lumber pre-drying in sawmills with an output range of 10,000 to 30,000 m^3/year depend very significantly on electricity costs. A range of US$0.06/kWh to $0.18/kWh in electricity price results in MW pre-drying cost between US$15.4 and $45.4/$m^3$. At an electricity price of $0.12/kWh, MW technology can provide savings between US$8.4 - $21.4/$m^3$ for sawmills with an output of 10,000 m^3, US$12.5 - $25.5/$m^3$ – 20,000 m^3/y and US$13.5 - $26.5/$m^3$ for sawmill output 30,000 m^3/y.

The cost of MW treatment of railway sleepers in a plant with an output 10,000 to 40,000 m^3/year are in the range of US$15-42/$m^3$ at electricity costs US$0.06/kWh to $0.2/kWh. At an electricity price range of US$0.12 to $0.20kWh, the cost benefit resulting from the production of MW treated sleepers will be in the vicinity of US$300,000 - $410,000 for sawmills with an output of 10,000 m^3/y. For sawmills with an output of 40,000 m^3/y, the cost benefit will be between US$1,640,000 to $2,040,000/year.

The costs of MW wood material processing are acceptable for industry and high economic advantages provide good opportunities for commercialization of the new MW technology.

economic efficiency; lumber; microwave wood modification; pulp; sleeper

Microwave drying of seeds and vegetable products: a viable option for Ecuador

28.07.2016 14:15 e-Poster Session (Show Room)

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Agriculture is a predominant activity in Ecuador, thus this sector is one of its main economic activities. seed conservation is an important issue within agricultural practices that requires a drying process for maintaining seed viability during storage. Moreover, seed conservation in Ecuador, is not only important from an agricultural point of view, but also as a strategy for conservation of the huge wild plant biodiversity in the country. In addition, drying is one of the main techniques for preserving agricultural and food products. Nowadays, Ecuador government is trying to promote the production of dehydrated fruits and vegetables. Since agricultural activity and biodiversity conservation are priorities for the Ecuadorian State [A], the research and development of new technologies to achieve an improvement of the drying process would be of great interest. Therefore, the main objective of this study was to assess the feasibility of applying microwave drying on seed conservation and dehydration of vegetable products for its potential use in Ecuador.

In this study, it was performed a literature review of the published research works on microwave drying technology focused on seed conservation and dehydrated fruits and vegetables, with special emphasis on those papers that analyze the quality of the final product.

The analysis carried out shows that microwave technology has been successfully applied to dry seeds and vegetables products due to its high energy efficiency and the increase of the drying rates, leading to a decrease in drying time [B][C][D][E]. However, this technology presents sometimes limitations for its application, such as the non-uniform temperature distribution which could generates hot spots that result in charring or burning the product [F]. Besides, the high heating rates may affect the quality of the final dried product. The essays conducted in this field report the possibility to overcome these disadvantages using lower power or controlling the drying temperature [G].

Taking into account the results obtained it can be concluded that the use of microwave technology has a great potential to improve the processes of seed conservation and production of high quality dehydrated fruits and vegetables, enhancing the energy efficiency of the drying process. Thus, the implementation of this technology would be very useful and viable for the agriculture sector of Ecuador.

microwave drying; seed conservation; dried fruits; vegetables

[A] SENPLADES (2013). Plan Nacional de Desarrollo /Plan Nacional para el Buen Vivir 2013-2017. Quito, Ecuador, Secretaría Nacional de Planificación y Desarrollo (SENPLADES).
[B] M. Maskan, Kinetics of colour change of kiwifruits during hot air and microwave drying. Journal of Food Engineering 48(2): 169-175, 2001.
[C] I. A., Ozkan, B. Akbudak and N. Akbudak, Microwave drying characteristics of spinach. Journal of Food Engineering 78(2): 577-583, 2007.
[D] R. Choudhary and D. G. Watson, Microwave drying kinetics and quality characteristics of corn. International Journal of Agricultural and Biological Engineering 6(1): 90-99, 2013.
[E] M. Hemis, R. Choudhary, Y. Gariépy and V.G.S. Raghavan, Experiments and modelling of the microwave assisted convective drying of canola seeds. Biosystems Engineering 139: 121-127, 2015.
[F] D. Wray and H. S. Ramaswamy, Novel Concepts in Microwave Drying of Foods. Drying Technology 33(7): 769-783, 2015.
[G] Z. Li, V.G.S. Raghavan and V. Orsat, Temperature and power control in microwave drying. Journal of Food Engineering 97(4): 478-483, 2010.

Selected topics on microwave application to green technology in our research group

POSTER 28.07.2016 14:15 e-Poster Session (Show Room)

Noboru Yoshikawa [A], Lee Chang Chuan [B], Manami Sunaka [B], Keita Kawahira [B], Shoji Taniguchi [A]

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In this paper, two projects on microwave application to environmental technology are reported. Rapid heating of Diesel Particulate Filter (DPF) by microwave was attempted. Porous stainless steel / glass composite body was fabricated and heated by microwave H-field. Combustion of carbon black was accomplished by an analysis of the gass flowing out.

Carbon reduction of TCP (tri-calcium phosphate) by microwave heating was conducted for recycling phosphorous, which was successfully obtained in vapor phase. The microwave reduction was accomplished at lower temperature than expected by thermodynamic calculation. The higher temperature and the longer heating resulted in the more reduction and vaporization, which is consistent with the normal kinetics. But an optimum heating rate has to be investigated when iron oxides exist.

Microwave; environmental technology; DPF; rapid heating; phosphorous recycling

Intensification of CO_2 capture processes using microwave heating

POSTER 28.07.2016 14:15 e-Poster Session (Show Room)

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[B] University of Edinburgh, School of Engineering, Institute for Materials and Processes, United Kingdom

Energy penalty of CO_2 capture from the flue gas generated at power plants is still of major concern, and more efficient capture methods need to be developed. Adsorption using solid sorbents is a promising solution to control CO_2 emissions from large - fixed sources, due to the potential of sorbents for high CO_2 capture capacity and selectivity, fast adsorption and desorption kinetics, good mechanical properties and stability after repeated adsorption - desorption cycles.

For the practical CO_2 separation we have chosen temperature swing adsorption (TSA), which involves cyclic adsorption - desorption processes where CO_2 is captured at post - combustion conditions and it is regenerated by increasing the temperature of the adsorption bed. However, the main drawbacks of TSA are the large amount of energy required for heating and cooling in each adsorption cycle, and the long heating/cooling times and therefore, the large equipment facilities needed. In conventional heating the heat transfer depends on the thermal conductivity of the packed bed, thus the temperature gradient along the packed bed is the result of the high thermal inertia or the limited heat transfer.

For these reasons, the design of more efficient adsorption - desorption processes is the focus of this work. To avoid the TSA drawbacks, we have developed an experimental bench - scale microwave - integrated fixedbed column to evaluate regeneration strategies by means of Microwave Swing Adsorption (MWSA).

The novelty of this process is based on the direct and selective volumetric heating of the adsorbent bed during the regeneration step, which is dependent on the adsorbent - microwaves interaction, and can avoid large thermal gradients, and consequently reduce the amount of energy applied to heat large columns of adsorbents. The main aim of using MWSA then is to minimize the overall capture process costs by reducing the energy consumption during the CO_2 regeneration.

This work presents the study of the interaction of microwaves with commercial CO_2 adsorbents, and its beneficial impact on the carbon capture process. Microwave swing regeneration of CO_2 from 13X zeolite, structured MCM - 41 silica, amorphous low - cost silica (FS) and alumina at different regeneration conditions was measured in this study. Results were evaluated based on the heating rate of materials after their exposure to microwaves, the resultant CO_2 desorption rates, both the amount and the purity of CO_2 recovered, the necessary regeneration times and the specific - energy consumption.

It was found that 13X zeolite is a strong microwave absorber, which has the capability to increase the temperature in a matter of seconds under the microwaves exposure. It achieved a complete regeneration after 6 min at 140℃. From this study we conclude that MWSA is a promising regeneration technology, which allows for very short regeneration times and has high potential to increase the overall separation efficiency with the subsequent energy penalty reduction of post - combustion CO_2 separation processes.

CO_2 capture; regeneration; desorption; microwave heating; adsorbents; zeolites

Topic 04: Enhanced chemical reactions

Microwave propagation in chemical reaction

PLENARY 26.07.2016 09:00 Main Room

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College of Electronics & Information, Sichuan University, Chengdu, 610065, China

Cutting-edge developments in microwave chemistry and material processing: latest applications of the microwave semiconductor generator

PLENARY 26.07.2016 09:20 Main Room

Satoshi Horikoshi
Dept. of Materials and Life Sciences, Faculty of Science and Technology, Sophia University, Japan.

A magnetron generator is generally used in the commercial microwave heating apparatus; on the other hand, uses of the semiconductor-based oscillator in microwave heating have significantly increased. The distinct features of the magnetron and the semiconductor generator in microwave heating will be compared in this presentation, and the advantages and practical applications of the semiconductor generator will be introduced.

The frequency of the microwave radiation from the magnetron generator in microwave cooking ovens is distributed over a large frequency range: from 2.25 to 2.60 GHz [A]. The distribution of the frequency changes depending on the characteristics of the microwave generator and equipment. By contrast, the semiconductor generator produces microwaves only within a very narrow frequency range of 2.4500 ± 0.0025 GHz. In the case of the magnetron generator, the microwave input power distribution reflects the widely dispersed frequency distribution (see table). Therefore, the output of the actual 2.45-GHz microwave is smaller than the input power. When using a semiconductor microwave generator on the other hand, the heating can progress efficiently because the microwave input power is concentrated at the frequency of 2.45000 GHz. The microwave can resonate better in a single-mode apparatus. The semiconductor microwave heating can then selectively progress because the *E*-/*H*-filled phases of the microwave can be concentrated by the synthesis of the microwave. Although the microwave output from the semiconductor generator is small, it is definitely possible to obtain a much higher input power by this synthesis of the microwave. The field of chemistry, as well as fields of material processing, food processing, and drying could potentially benefit by utilizing this technology. The disadvantages of the semiconductor generator, however, have always been the price and the available maximum power. Nonetheless, semiconductor generators with the maximum power of several hundred Watts and highly accurate microwave frequencies are recently available at prices very similar to magnetrons: these prices are falling exponentially.

	Magnetron	Semiconductor
Applied power	30 W～kW	～3 kW
Weight of the power supply	6 kg (for 1 kW)	0.5 kg (for 1 kW)
Applied voltage	1 kV (for 1 kW)	0.05 kV (for 1 kW)
Electric power change efficiency	70－80 %	50－70 %
Frequency control	No	Yes
Lifetime	500－1000 hrs	20 years
Price	Cheap	Expensive
Use environment	Weak in vibration	Weak for high temp. and reflected MW

▲Comparison of the microwave's magnetron and semiconductor generators

semiconductor microwave generator; GaN; microwave oven; phase control

Creating ultra-high temperature ceramic matrix composites by microwave or RF assisted chemical vapour infiltration

PLENARY 27.07.2016 09:00 Main Room

John Binner
School of Metallurgy and Materials, College of Engineering & Physical Sciences, University of Birmingham, Edgbaston, Birmingham, B15 2TT, UK

There is an increasing demand for advanced materials with a temperature capability of well over 2000℃, in highly corrosive environments and whilst subject to intense heat fluxes and mechanical stress associated with vibration, for aerospace and other applications. The interaction of environmental conditions together with the requirement that dimensional stability is maintained makes the selection of suitable materials extremely challenging. This paper discusses the design, development, manufacture and testing of a new class of Ultra-High Temperature Ceramic Matrix Composites (UHTCMCs). Based on C fibre preforms enriched with ultra-high temperature ceramics (UHTC) via both powder impregnation and then microwave or RF assisted chemical vapour infiltration, the composites are intended for application in severe aerospace environments.

Encounter of microwave chemistry and microwave engineering

PLENARY 28.07.2016 09:00 Main Room

Naoki Shinohara
Research Institute for Sustainable Humanosphere (RISH), Kyoto University , Japan

Microwave assisted heterogeneous catalysts, how to deposit energy and measure temperature

ORAL 26.07.2016 18:00 Session B (Room 305)

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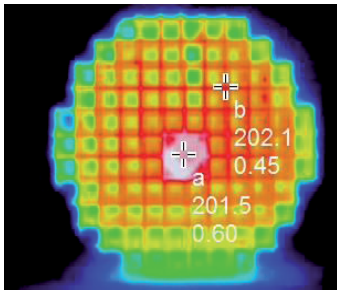
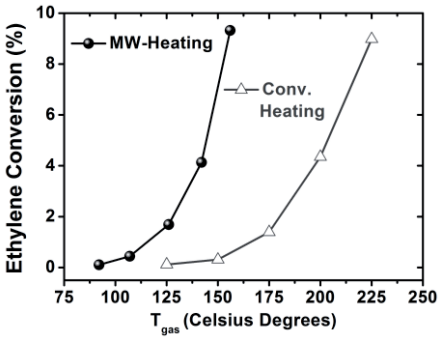
The enhancement of chemical reactions using microwaves has been widely exploited in organic liquid synthesis in the last 30 years. It has been demonstrated that in many cases the so called "microwave effect" does not exist and the observed effects were purely thermal and not related to the microwave field [A]. In the case of heterogeneous catalysts many of the studied systems include endothermic reactions thermodynamically limited by equilibrium. Also in this case, due to a higher temperature achieved using microwaves, an apparent equilibrium is established and thus thermodynamic equilibrium "could be overcome". It is evident that an accurate temperature measurement is of paramount importance to describe well microwave assisted reaction systems.





◀Thermal image of a cordierite monolith impregnated with MW absorbing catalyst (left), and Ethylene conversion vs. temperature for microwave and conventional heated systems in the ethylene epoxidation reaction (right).

MW heating of a solution containing polar molecules can be reasonably well explained as a consequence of dipole rotations. However in solids, where there are no dipoles able to rotate freely, heating is explained as the result of different categories of polarization induced by the electric field, namely electronic, atomic, dipolar and interfacial polarizations. Even for relatively simple systems, this may lead to interplay of influences that are not easily visualized in terms of a single physical model [B].

In this work we present two important aspects related to heterogeneous microwave heated catalytic systems which are the measurement of the temperature and the study and selection of microwave absorbing catalyst. This includes electron correlated materials where metal to insulator transitions, IMT could be induced by the electromagnetic field.

heterogeneous catalyst; temperature measurement; correlated materials

[A] Herrero, M.A. et al., Nonthermal microwave effects revisited: On the importance of internal temperature monitoring and agitation in microwave chemistry, J. Org. Chem. vol 73, no 1, pp. 36-47, 2008.
[B] Gracia, J. et al-, Nano-heaters: New insights on the outstanding deposition of dielectric energy on perovskite nanoparticles, Nano Energy, vol 20, pp. 20-28, 2016.

Efficient reduction of copper oxide with carbon using microwave local heating

ORAL 26.07.2016 18:15 Session B (Room 305)

Naoto Haneishi, Shuntaro Tsubaki, Masato M. Maitani, Eiichi Suzuki, Satoshi Fujii, Yuji Wada
Tokyo Institute of Technology, Japan

Microwaves heat the materials by loss of vibrated electromagnetic field energy. When the packed bed is exposed to microwaves, distributions of electromagnetic field depending on a solid filling state are formed in the sample layer. These distributions determine the distribution of temperature in the packed bed. We focus on the phenomenon that electric field of microwaves is concentrated at the contact points of dielectric particles and build up a hypothesis that microwave heating can accelerate the chemical reaction proceeding at the contact point of dielectrics.

Carbon reduction of copper oxides under microwave irradiation was observed by using thermogravimetry. Reduction of copper oxide under microwave heating occurred 250 degrees lower than conventional heating. Temperature distribution in the packed bed was analyzed by the electromagnetic and thermal flow simulation methods.

microwave heating; carbon reduction; thermogravimetry; electromagnetic simulation

Microwave-enhaced dehydrogenation of 2-propanol over magnite catalyst

ORAL 26.07.2016 18:30 Session B (Room 305)

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Microwaves often exhibit enhanced reaction rate and product selectivity due to non-equilibrium local heating of the microwave-susceptible solid catalyst [A][B]. This paper demonstrates effects of microwave non-equilibrium local heating on dehydrogenation of 2-propanol over magnetite (Fe_3O_4) catalyst using a fixed-bed flow reactor. The microwave reaction was conducted by using the high power amplifier module (HPA) microwave generator equipped with an elliptical applicator. Temperatures at the core and surface of the catalyst bed were monitored with fiber-optic and IR thermometers, respectively. Production of acetone from 2-propanol was monitored by an on-line GC (TCD). Microwave

irradiation exhibited higher acetone yield than conventional heating using a mantle heater by 11.5-fold with 90 – 95% of selectivity. The activation energies for the microwave reaction were 34 kJ/mol (core) and 41 kJ/mol (surface) while that for the conventional heating reaction was 118 kJ/mol. The results indicated that dehydrogenation of 2-propanol was facilitated by the uneven temperature distribution in the catalyst bed under microwave irradiation. Uneven temperature distribution in the catalyst-bed was also confirmed by simulating temperature distribution using the finite element method (COMSOL).

microwave non-equilibrium local heating; fixed-bed reactor; magnetite; dehydrogenation; 2-propanol

[A] Fujii, et al., Methanol decomposition reaction using Pd/C as solid catalyst under highly precise microwave irradiation, Microwave Symposium Digest (MTT), 2012 IEEE MTT-S International.
[B] Ferrari et al., Microwave-specific effects on the equilibrium constants and thermodynamics of the steam-carbon and related reactions, The Journal of Physical Chemistry C, 118, 9346-9356, 2014.

Insight into microwave-driven catalytic reactions: non-equilibrium local heating and acceleration of electron transfer

26.07.2016 18:45 Session B (Room 305)

Yuji Wada[A], Shuntaro Tsubaki[A], Takeo Yoshimura[A], Masato Maitani[B], Dai Mochizuki[C], Funminao Kishimoto[A], Naoto Haneishi[A], Ryo Sasaki[A]

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[C] Interdisciplinary Cluster for Cutting Edge Research, Center for Energy and Environmental Science, Shinshu University, 3-15-1, Tokida, Ueda City 386-8567, Japan

Microwaves often exhibit specific effects such acceleration of reaction rate and increased selectivity of the products. Although there are numbers of reports regarding “microwave effects” in organic and inorganic chemistry, only a few papers have dealt with their fundamental mechanisms. In this paper, we will demonstrate two examples of microwave effects with different mechanisms; non-equilibrium local heating and acceleration of electron transfer. The non-equilibrium local heating was kinetically studied by using carbon-filled core-shell zeolite catalyst [A]. The carbon-filled core exhibited selective heating under microwave irradiation, resulting in accelerated oxidation of 1-phenylethanol in the micropores of the shell zeolite twice as fast as the conventional heating at the same temperature. This mechanism can be recognized as “thermal effect”. In contrast, acceleration of electron transfer was observed for photoreduction of an electron acceptor on CdS quantum dots [B]. Photoluminescence measurements from the excited-sate CdS revealed that photo-induced electron transfer was accelerated under microwave irradiation by 3.5-fold. This result proved the existence of another microwave effect, “non-thermal effect”.

thermal effect; non-thermal effect; core-shell zeolite; electron transfer
[A] D. Mochizuki, et al., Catalytic reactions enhanced under microwave-induced local thermal non-equilibrium in a core-shell, carbon-filled zeolite@zeolite, Journal of Catalysis, 323, 1-9, 2015.
[B] F. Kishimoto, et al., Microwave-enhanced photocatalysis on CdS quantum dots – Evidence of acceleration of photoinduced electron transfer, Scientific Reports, 5, 17, 11308, 2015.

Catalytic revalorization of glycerol to fuel additives. Microwave effect in catalysts and in reaction

POSTER 28.07.2016 14:15 e-Poster Session (Show Room)

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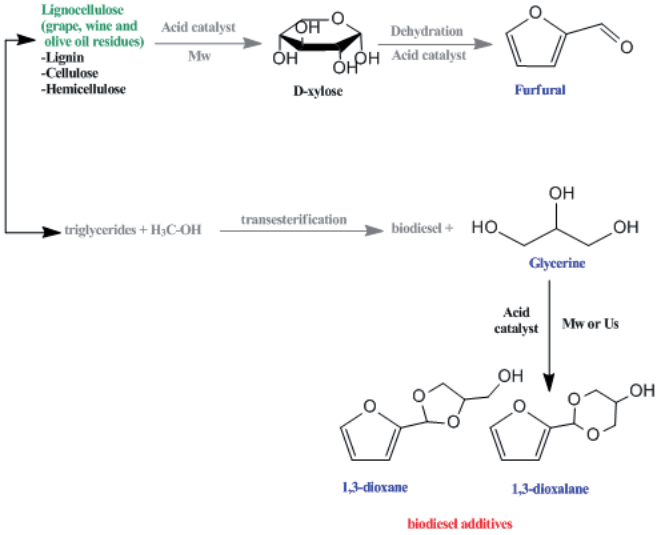
Currently, in the petrochemical industry, crude oil is fractionated and refined to produce various grades of liquid transportation fuel. Additionally, hydrocarbon feedstocks are functionalized to produce intermediates and, particularly, chemicals. The analogue concept of biorefining would be similar in scope, with the difference that biomass would be used as a renewable source. Conversion of biomass into functionalized platform molecules, such as sugars (glucose, xylose), polyols, furans (furfural, 5-hydroxymethylfurfural) and acids is unique to hydrolysis-based methods and allows for the production of a wide range of fuels and chemicals [A]. Appropriate upgrading strategies using catalytic technologies should be developed to obtain high-added value products from biomass. Thus, the catalytic acetalization of furfural with glycerol, a byproduct generated during biodiesel manufacture in high amounts, is an attractive process for the production of a mixture of cis and trans isomers of five- and six-membered furans named 1,3-dioxalane and 1,3-dioxane, respectively. These products can be used as biodiesel additives since they can improve the biodiesel viscosity and cold properties.



The aim of this work was to study the effect of using microwaves for catalysts preparation and in catalysis. Microwaves can decrease the synthesis time and temperature, provide different properties in the final catalysts [B], and enhance the activity results to the products of interest.

To meet this goal, 10 micro- and meso-structurated acid catalysts were prepared. Beta, mordenite, montmorillonite and SBA-15 were sulfonated using conventional heating (SBR, SMR, SMontR, SSBA-15R) and with microwaves (SBMw, SMMw, SmontMw, SSBA- 15Mw). Additionally, Na-ZSM-5 was exchanged with Ga3+ by conventional heating (Ga-ZSM-5), and

with microwaves (Ga-ZSM-5-Mw). The catalytic reaction was carried out for all catalysts by conventional heating (glycerol: furfural ratio 1:1, 40℃, 2 h, 5 wt % catalyst) and with microwaves for catalysts SBMw, SMMw and SMontMw at the same reaction conditions.

Microwaves allowed us the incorporation of higher amounts of sulphonic groups in SBA-15 and montmorillonite catalysts. The catalytic tests using microwave heating involved a certain loss of conversion, compared to the use of conventional heating, but an increase in the dioxalane selectivity was observed accompanied by a decrease of the selectivity to by-products.

microwaves; biomass; biodiesel additives; acid catalysis.

[A] R.A. Sheldon, Green and sustainable manufacture of chemicals from biomass: state of the art, Green Chem. vol. 16, 950 - 963, 2014.
[B] M. D. González, Y. Cesteros, J. Llorca, P. Salagre. Boosted selectivity toward high glycerol tertiary butyl ethers by microwave - assisted sulfonic acidfuncionalization of SBA - 15 and beta zeolite, J. Catal. vol. 290, pp. 202 - 209, 2012.

Microwave assisted Michael addition of phenols or anilines in the presence of DMAP

28.07.2016 14:15 e-Poster Session (Show Room)

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In 2005, Michael addition between phenol and acrylonitrile was reported by Al-Awadi et al. using aqueous benzyltrimethylammonium hydroxide as a catalyst [A]; otherwise, the reported reaction time exceeded 20 h. The same Michael addition was also reported by Gunnoe et al. using Cu(I) catalysts [B], however, the long reaction time (40 h) of this reaction was problematic. We are interested in organic reactions under non-conventional conditions such as high pressure and microwave irradiation. We reported that the 1,2-addition reaction between acetophenone and trimethylsilyl cyanide (TMSCN) occurred under microwave irradiation conditions but did not occur under high–pressure conditions [C]. We also reported that microwave irradiation might facilitate the conjugate additions of chalcones and TMSCN without additives or solvent [D]. Sometimes, microwave irradiation methods are useful to shorten reaction time. Recently, we reported our attempts of microwave irradiation conditons to the Michael addition between phenols and acrylonitrile; the reaction rates were strongly accelerated and Michael adducts were rapidly produced compared to the abovementioned reports [E]. Further our investigations of microwave assisted Michael addition between phenols and acrylonitrile, we recognized that the reaction proceeded smoothly in the presence of a catalytic amount of 4-dimethylaminopyridine (DMAP). The reactions using phenol, 4-methylphenol or 4-methoxyphenol as a Michael donor proceeded very rapidly to obtain corresponding Michael adducts within 4 h over the yields of 80%. When we applied the conditions for the

Michael addition between anilines and methyl acrylate, the reactions also proceeded rapidly; for example, using aniline or 4-methylaniline as a Michael donor, both reactions were completed within 30 min to yield corresponding Michael adducts. These results suggest that DMAP is one of the best catalysts for Microwave assisted Michael addition.

michael addition; microwave irradiation; phenol; aniline

A S. A. Al-Awadi, et al., Kinetics and mechanism of thermal gas-phase elimination of β-substituted carboxylic acids; Tetrahedron, vol. 61, no. 24, pp. 5769-5777, 2005.
B C. Munro-Leighton, et al., Anti-Markovnikov N-H and O-H Additions to Electron-Deficient Olefins Catalyzed by Well-Defined Cu(I) Anilido, Ethoxide, and Phenoxide Systems; J. Am. Chem. Soc. Vol. 128, no.5, pp. 1446-1447, 2006.
C H. Iida, et al., Highly efficient cyanosilylation of aldehydes and ketones under microwave, solvent-free, and Lewis acid-free conditions; Synth. Commun., vol. 37, no. 11, pp. 1801-1805, 2006.
D H. Iida, et al., An efficient conjugate hydrocyanation of chalcones and related enones with TMSCN under solvent- and additive-free microwave conditions; Tetrahedron Lett., vol. 48, no 11, pp. 2037-2039, 2007.
E H. Iida, et al., Efficient and rapid synthesis of phenolic analogues of 4-phenylbutanoic acid using microwave–assisted Michael addition as a key reaction; Synth. Commun., 2016, *in press*.

Microwave power dependence corresponding to temperature dependence on enzymatic reactions

POSTER 28.07.2016 14:15 e-Poster Session (Show Room)

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On enzymatic reaction, we had assumed the microwave power dependence was conceivable to be related physical properties of substrate. In this study, we estimated the relationship between physical properties of substrate and microwave power dependence. As an experimental method, the hydrolysis reaction of p-nitrophenyl acetate was carried out by using lipase from Candida rugosa. Some kinds of carboxylic acid and p-nitrophenol ester were selected as a substrate to investigate the relationship between the microwave power dependence and substrate properties. We have already revealed the microwave power dependence in microbial cultivation under microwave irradiation. This result also means the presence of microwave power dependence of enzyme reaction in the microorganism. In this study, we intended to make clear the microwave power dependence in enzymatic reaction. As an example, the Hydrolysis reaction was examined with using lipase from Candida rugosa as model reaction. The reaction temperature and microwave power was controlled with using reactor attached cooling jacket exactly. Then, we examined the microwave power dependence in enzyme reactions, and the tendency of the microwave power dependence regarding the reaction temperature. As a result, it was found that the Michaelis constant（Km）and the maximum initial velocity（Vmax）of enzymatic reaction vary for microwave power. In addition, it was also found that the microwave power dependence changes in the reaction temperature. This phenomenon could be considered to be related to the specific heating principle of microwave heating. In microwave

irradiation, highly polar molecule causes the rotational motion. Thus, it is considered to increase collision frequency of molecules. Therefore, it will be predicted that the microwave power dependence of enzymatic reaction is involved in collision frequency between enzyme and substrate. Accordingly, it will be necessary to examine the microwave power dependency in more low-temperature condition.

enzymatic kinetics; invertase; lipase; microwave power dependence

Enhanced chemical vapour infiltration of high temperature ceramic matrix composites (HT-CMCs)

28.07.2016 14:15 e-Poster Session (Show Room)

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The ability of HT-CMCs to operate in hostile aero-thermo-chemical environments without compromising on structural integrity, whilst keeping mass at a premium, makes them suitable candidates for future aerospace propulsion and manoeuvrability systems. Fully dense components are currently manufactured by chemical vapour infiltration (CVI) using isothermal heating and multiple machining stages to prevent residual porosity forming. As a result, the processing time is 2 to 3 months and components are very expensive. Microwave (MW) and radio frequency (RF) energy has been proposed to offer a potential solution by creating an inverse temperature profile, so densification starts on the inside and proceeds outwards. This avoids the closure of surface porosity facilitating reactant gases transport throughout the structure. Subsequently, it is expected that using the enhanced CVI methods manufacturing time can be decreased to about 72-96 hours.

Topic 05: Equipments

On proper use of magnetron antenna probes

ORAL 28.07.2016 12:00 Session B (Room 305)

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Magnetron antenna probes are devices that enable low-power (cold) measurement of the Rieke diagram-related load reflection coefficient ordinarily perceived by the installed magnetron. This knowledge is particularly important when designing installations without circulators protecting magnetrons. Antenna probes are typically accompanied by conventional instructions for their use and a data table characterizing the probe. While the conventional procedure uses a vector network analyzer (VNA), it does not fully exploit the powerful capabilities of present-day VNAs. In addition, the procedure is laborious, prone to errors, does not allow real-time swept measurements, and the results are biased by a systematic error equal to the residual reflection coefficient of the probe.

This paper will present the basic theory of antenna probes, elucidate the conventional method of their use, and propose an improved procedure, including needed accessories, consisting of a reference launcher (as stipulated in magnetron datasheets) and a set of at least three offset calibration shorts of the launcher waveguide type.

The procedure is essentially a standard vector reflectometer calibration at the waveguide output of the reference launcher with installed probe as shown in Fig. 1.

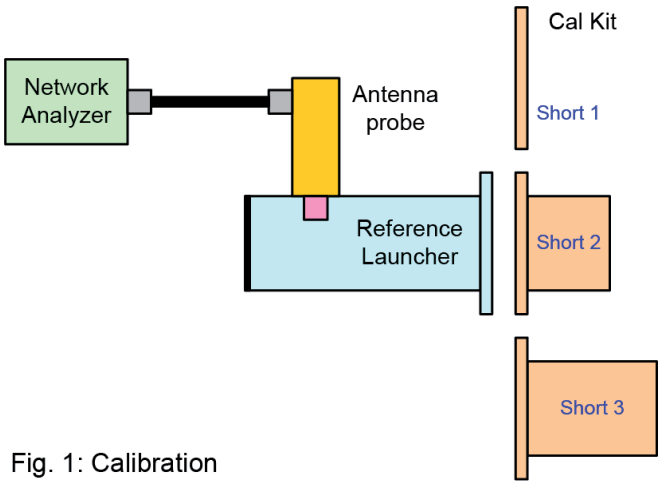

Fig. 1: Calibration

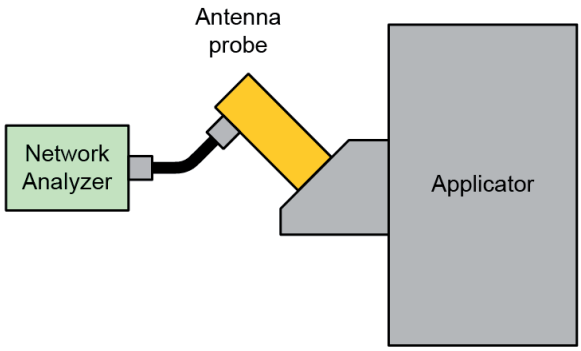

Fig. 2: Application

Such calibration will enable error-free, real-time, swept observation of Rieke reflection coefficient of an arbitrary installation (Fig. 2). We will illustrate this case on a measurement example and compare the two methods. We will also discuss the proper interpretation of the swept Rieke reflection coefficient measurement results.

Rieke diagram; magnetron; magnetron antenna probe; vector reflectometer calibration

Microwave wood modification technology and equipment for its commercialization

28.07.2016 12:15 Session B (Room 305)

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The use of microwave (MW) technology is growing in timber industries. This arises because of the high efficiency of converting electricity into MW energy; energy savings associated with rapid in depth heating of materials; specific interactions that can be achieved between microwaves and materials; radical acceleration of technological processes; reductions in MW equipment costs and improvements in the reliability of industrial MW equipment.

The new technology of MW wood modification is based on the supply of high intensity MW power, at frequencies 0.922 and 2.45 GHz. Such power induces significant changes to the microstructure of wood creating a dramatic increase in wood permeability. A number of commercial applications have been developed based on increased the wood permeability including: the treatment of refractory wood species with preservatives, rapid drying of hardwoods, relief of growth and drying stresses in timber, the manufacture of the new wood materials Torgvin and Vintorg, modification of logs, sawn timber and woodchips for pulping.

MW timber modification is a continuous conveyor process and occurs where the green timber or logs move through a MW field in the applicator which focuses high intensity MW energy into a limited volume of wood. The main MW processing parameters comprise: MW frequency, specific power applied to the wood, energy supplied to wood, MW applicator configuration, timber feed speed through MW applicator, and air flow parameters (temperature and speed). These rational parameters have been developed to provide required levels of MW wood modification. The specific power applied to the wood at 2.45 GHz ranged from 5,000 to 135,000 kW/m^3 and at 0.922 GHz ranged from 5,200 to 26,000 kW/m^3. The MW energy for timber modification supplied to the wood ranged from 216 to 1550 MJ/m^3 (60 to 430 kWh/m^3). A new class of special MW applicators for high intensity energy supply to the wood was developed. Semi- commercial and commercial plants for wood modification for different purposes were designed and manufactured in Australia and China.

MW wood modification technology provides significant material and energy savings and improves both economic and environment performance of a very traditional industry. The costs of MW timber processing for different applications are in the range of AU$ 22-69/m^3. This low cost technology should find wide commercial appeal and use in the timber, biocomposite and pulping industries.

microwave applicator; microwave wood modification; pulp; timber

Topic 06: Industrial applications, manufacturing, processing controls

Bridging gaps in microwave technologies for industrial production of safe foods

27.07.2016 09:20 Main Room

Juming Tang
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Microwave-assisted extraction in natural products

27.07.2016 09:40 Main Room

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Next generation energy efficient mineral processing achieved through high intensity microwave heating

27.07.2016 10:00 Main Room

Chris Dodds
University of Nottingham, United Kingdom

High-power industrial microwave applications and market trends

27.07.2016 11:15 Session B (Room 305)

Jens Hofman
Muegge, Germany

High-power industrial microwave heating:

Microwaves facilitate targeted heating of a large variety of materials used in industry, be it homogeneous heating from the core to the surface or focused heating of a selected area or volume. This is a big advantage compared to conventional heating that is restricted to heating of the surface of materials. New developments particularly in power supply technology are aimed at meeting the challenges of industrial microwave heating.

An increasing number of industrial heating processes are sensitive to overheating and therefore require specific control technologies. In order to avoid overheating, microwave power supplies with extremely short response times are required. These short response times cannot be met by linear power supplies. Therefore, the new switch mode power supply (SMPS) technology is gaining increasing market share.

State-of-the-art single microwave heating units provide microwave power in the range of 300 W up to 30 kW at 2.45 GHz and between 5 kW and 100 kW at 915 MHz. In the near

future, single 915 MHz microwave heating units are expected to supply a maximum power of 125 kW. Operation of several microwave heating units in parallel easily meets the requirements of industrial microwave heating processes. Applications can be found in vacuum drying, sterilization and pasteurization in the food industry, in manufacturing of multilayer timber panels in the woodworking industry, in focused activation of chemical reactions in the pharmaceutical industry and in the manufacturing of insulating materials for sound absorption and for energy saving, respectively, in the construction materials industry for example.

High-power industrial microwave plasma technology:

Microwave-induced plasmas are another large application area of industrial microwave technology. At low pressure conditions, non-equilibrium plasmas are formed by microwave plasmas. They are characterized by the predominant excitation of electrons, i.e. the formation of "hot" electrons, while the heavy ions and neutral particles in the plasma stay almost at room temperature. As a consequence, plasma surface treatment of even thermal sensitive materials is feasible. Additionally, high electron densities inducing high densities of radicals are achievable by microwave plasmas. As a consequence, high etching and deposition rates, respectively, can be obtained.

The Remote Plasma Source (RPS) operated at low pressure is characterized by a high radical density outside of the plasma chamber. As a result, the RPS is applied for e.g. selective, fast and highly efficient plasma etching of semiconductor and organic materials, in particular. RPS etching rates of more than 200 μm/h can be achieved for organic materials like SU-8 photo resist. Duo-Plasmaline plasma sources are another type of microwave plasma sources providing highly efficient and fast plasma etching of different kinds of materials.

However, their major field of application is plasma deposition. Prominent examples are a) scratch resistant layers deposited by high-rate plasma processes with deposition rates higher than 50 μm/min for transparent polymers used in the construction material and in the automotive industry, b) highly impermeable gas and water vapor barrier films in the nano-range required for example in food packaging as well as for encapsulation of thin-film solar cells and even organic light emitting diodes (OLEDs), c) passivation layers in photovoltaics as well as d) artificial diamond for a variety of applications.

Microwave power supplies based on the newly developed switch mode power supply (SMPS) technology can easily be pulsed to enlarge the parameter space in order to improve the quality of the surface modified in the plasma process.

Atmospheric pressure microwave plasmas can be used to form both non-thermal plasmas like at low pressure and thermal plasmas. While the non-thermal plasma of the Micro Plasma Jet is applied e.g. for efficient inside cleaning of long but narrow cannulas, the thermal plasma of the an Atmospheric Plasma Source (APS) is very well suited for

abatement of waste gas containing fluorinated and chlorinated volatile organic compounds (VOCs), for rapid activation of different kinds of surfaces and for deposition of amorphous layers for application in photovoltaics for example.

Experimental comparison of microwave and radio frequency tempering of frozen beef

27.07.2016 11:30 Session B (Room 305)

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The objective of this study was to experimentally compare the microwave and radio frequency tempering of frozen beef in terms of tempering rate and uniformity. To do this, a block of frozen lean beef (~2.4 kg) was tempered from an initial temperature of -24℃ to between -5 and 0℃ both in a specially designed microwave system (915 MHz, 5 kW maximum power) and a radio frequency oven (27.12 MHz, 2 kW maximum power). Temperature at four different locations during tempering was recorded by using a signal conditioner and fiber optic probes. For microwave tempering, the block of frozen beef were placed in the center of the cylindrical cavity of the system. Microwave tempering process was conducted at a power setting of 500 W. For radio frequency tempering, the block was placed in between the electrodes of the system. Electrode gap was adjusted to be 160 mm.

Time needed for temperature increase from the initial to between -5 and 0℃ at all four locations where the fiber optics were inserted was about 20 minutes for both treatments. However, radio frequency tempering was found to be more uniform than microwave tempering for the experimental conditions studied. Although the temperatures that were measured at four locations during microwave tempering seemed to be closer to each other, there were parts that apparently overheated (run-away heating) resulting in non-uniform tempering which, in fact, led to partial cooking of the beef. Radio frequency tempering method provided a much more uniform tempering when compared to microwave tempering with no overheated/cooked parts. Uniformity of tempering by microwaves may be improved by using a turntable within the cavity of the microwave system. If power applied during microwave tempering is increased in addition to employing a turntable, a more rapid tempering along with increased uniformity can be achieved.

frozen beef; tempering; microwave; radio frequency

Microwave tempering of frozen block of shrimp in a cylindirical cavity

27.07.2016 11:45 Session B (Room 305)

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The objective of this study was to experimentally evaluate the performance of a specially designed microwave system (915 MHz, 5 kW maximum power) in terms of tempering rate and uniformity. To do this, a block of frozen shrimp (~2 kg) was placed on a turntable inside the cylindrical cavity of the system and tempered from an initial temperature of -22℃ to between -5 and -3℃ at two different power settings (500 and 1000 W). Temperatures at four different locations were recorded during tempering by using a signal conditioner and fiber optic probes. Surface temperature distribution of the shrimp block before and after tempering was also measured using a thermal camera.

Time needed for temperature increase from the initial to between -5 and -3℃ at all four locations where the fiber optics were inserted was about 10 and 4 minutes at power settings of 500 and 1000 W, respectively. Temperature increase at the center was found to be the fastest, since the center of the block was directly facing the waveguide throughout the experiment. Temperature increase at the corner was the slowest, since the corner of the block was moving in and out of the path of the waveguide. Temperature at the corner also fluctuated more for the same reason. No overheating was observed for either power setting. The greatest standard deviations of temperature readings including all four fiber optic probe locations during tempering at 500 and 1000 W were 2.8 and 3.8℃. Temperature distribution was also very uniform at the surface of the block after tempering as shown by the thermal image. The results showed that rapid tempering of frozen foods can be achieved with acceptable uniformity in the specially designed microwave system.

frozen shrimp, tempering, microwave, cylindrical cavity, turntable

Drying of fruit and vegetables with the use of microwaves under low pressure

27.07.2016 12:00 Session B (Room 305)

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Promis-Tech sp. z o.o., Wrocław, Poland

The aim of the process of drying is to dehydrate the material while preserving its aroma values and its valuable active biological elements. In general, except for the sublimation method, it can be assumed that the quality of dried product is determined by three crucial elements: the duration of drying, the temperature to which the material being dried is heated up in the process, and the heating uniformity.

Uniquely favourable characteristics of dried product can be obtained through the use of the microwave method in vacuum drying process. Microwave-vacuum drying is superior to other methods in terms of dried products' structure, flavour, colour and biological active compounds contents. Furthermore, the so-called "volumetric heating phenomenon" makes this particular method energy-saving.

The article describes the mechanism of drying and the original structures of devices for drying biological material in lowered pressure, supported by microwave energy. It presents both laboratory structures intended for a wide range of testing of the drying processes of various biological materials, as well as some installations operating on industrial scale. The scaling-up from 2 kW of microwave power laboratory systems to 24 kW of microwave power industrial, complete technological lines is presented.

Apart from the description of the structure of the developed and manufactured microwave devices and technological lines, there are presented results of some tests of process parameters and instances of specific applications of such driers.

microwave application; microwave drying; low pressure drying

[A] Sunjka P.S., T.J. Rennie. 2004. "Microwave–convective and microwave–vacuum drying of cranberries: A comparative study". Drying Technology 22(5): 1217-1231.
[B] Grześkowiak P. R. Parosa. 2013. "The process of drying fruits, vegetables and herbs by microwaves in low pressure". Eurodrying Conference. Paris.
[C] Konopacka D., R. Parosa. 2015. "Ultrasound ad Microwave Hybrid Device for drying solid materials of high adhesion". Nord Drying Conference, Gdańsk.

Microwave-vacuum drying of sage leaves

27.07.2016 12:15 Session B (Room 305)

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The aim of this study was to investigate the effect of drying parameters, namely microwave power and vacuum level on quality characteristics of dried sage. To do this, sage leaves were dried in a laboratory scale microwave-vacuum dryer (2.45 GHz, 1.8 kW maximum power) from a moisture content of 71.6% (w/w) to below 10%. Three different levels of microwave power (90, 180 and 270 W) and system pressure (40, 75 and 110 mBar) were tested. Sage leaves were also dried conventionally (hot air). For hot air drying, air temperature used was 60℃. Quality characteristics of the leaves dried by two different methods were compared.

It was reported in the literature that the time required for hot air drying of sage leaves at 60℃ to a moisture content of 9.2% was about four hours [A]. According to the preliminary results of the present study, 15-20 minutes was adequate to reduce the moisture content of fresh leaves to below 10% when microwave-vacuum drying method was used. Color change upon drying was also investigated. Microwave-vacuum dried sage leaves were found to be greener than those dried by hot air. According to these results, microwave-vacuum drying not only reduces the drying time, but also helps better retain the color of fresh leaves. The study showed that microwave-vacuum drying holds great potential in

terms of reducing energy costs associated with drying as well as providing a dried product with superior quality and nutrient content.

Sage; drying; microwave; vacuum; quality characteristics

[A] P. R. Venskutonis, Effect of drying on the volatile constituents of thyme (Thymus vulgaris L.) and sage (Salvia officinalis L.), Food Chemistry, Vol. 59, no. 2, pp. 219-227,1997.

Microwave assisted bonding of synthetic leather to plastic substrates

ORAL 27.07.2016 16:00 Session B (Room 305)

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The lamination of substrates with natural and/or artificial coatings is commonly used in furniture, automotive, avionic and sport-tools industries. For lamination hot melt adhesives are widely used which are typically activated by resistive heating up to 80 to 140°C. This requires a heat transfer through the coating and/or the substrate. As a consequence all materials need to be heated to the curing temperature of the hot melt glue that takes significantly more time and energy as required for curing of the glue only. Furthermore the activation temperatures are limited by the thermal stability of the adherends. To make the process more flexible and to decrease its energy consumption and shorten processing time, the replacement of conventional heating by a selective microwave heating is investigated in the present paper.

The selective heating of the adhesive which is located in between the coating and the substrate is possible if the adherends are enough transparent for microwaves and the adhesive is a good microwave absorber. Moreover, for successful processing a microwave applicator is required that provides a sufficient and controllable microwave power density within the materials. In the present study the artificial leather and plastic substrates were chosen, which are relevant for automotive industry for decoration of interiors. The adhesive used is the reactive hot melt blended with carbon particles. A standard HEPHAISTOS [A] oven was used as a microwave applicator. The achieved results are summarized as follows:

The dielectric characterization of the hot melt as well as the artificial leather and substrate was performed within the working temperature range.

The absorbed microwave power was simulated in CST MWS by solving the wave equation for e/m waves at 2.45 GHz for a geometry of the HEPHAISTOS oven and the measured permittivity of materials.

The temperature fields within a substrate-glue-coating sandwich structure were simulated by solving the transient heat equation in COMSOL Multiphysics. The feasibility to reach a

desired temperature profile with a gradient of 40℃ within such a sandwich structure was verified in the model.

Experiments on the microwave assisted bonding of synthetic leather to plastic substrates in HEPHAISTOS oven were performed. Both carbon content and heating scenario were varied to find optimal conditions. A successful bonding was demonstrated and the quality of adhesion was checked in accordance with the standards in the lamination industry.

lamination; microwave bonding; electromagnetic and heat transfer modelling; hot melt

[A] http://www.voetsch-ovens.com/en/products/industrial_microwave_system/schunk01.c.59509.en

Microwave calcination of clays

ORAL 27.07.2016 16:15 Session B (Room 305)

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The industry is demanding new processes of calcinations of clays, particularly for low and medium production amount, being microwave processing a promising technology for that. However, clay minerals suffer very significant dielectric changes during microwave heating that must be taken into account.

It was evidenced during heating in an industrial microwave kiln that the material showed significant absorption changes that affect the performance of the microwave applicator.

To evaluate the changes during heating, samples of clays rich in kaolinite and smectite were selected and their dielectric properties during microwave heating were determined. Mineralogical analysis of clay minerals and associated minerals were made using X-ray diffraction at several temperature of treatment, using also DSC and DTA to evaluate changes during heating.

Complex permittivity (ε) was measured simultaneously during microwave heating using a controlled heating rate in a dual-mode cylindrical microwave cavity working near 2,45 GHz [A]. The use of two independent modes allows heating the sample with microwave energy and at the same time measuring the dielectric properties without interferences between both modes. The absorbed power at each temperature is also calculated from the ratio between the power supplied by the source and the return losses in a frequency range around the resonance frequency of the heating mode. With this equipment, the samples are characterized under the same conditions encountered in the industrial process.

Determination of dielectric constant (ε') and loss factor (ε'') during heating was correlated with the mineralogical changes. Different stages that correspond to variations in humidity and mineral transformation were identified, as follows: drying (humidity loss), structural

water loss, dehydroxylation (hydroxyl group loss) and crystalline change (mullite formation and others). In presence of carbonate minerals, decarbonation also took place.

Dielectric changes evaluated and its correlation to mineralogical changes were used to optimize the industrial processing.

microwave kiln; calcination; clays; dielectric properties

A J.M. Catalá-Civera, Canós, A.J.; Plaza-González, P.; Gutiérrez, J.D.; García-Baños, B.; Peñaranda-Foix, F.L., Dynamic Measurement of Dielectric Properties of Materials at High Temperature during Microwave Heating in a dual mode Cylindrical Cavity in Microwave Theory and Techniques, IEEE Transactions on , vol.63, no.9, pp.2905-2914, Sept. 2015.

Microwave assisted reduction

ORAL 27.07.2016 16:30 Session B (Room 305)

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Microwave applications to primary metallurgy is a growing field of interest, with an increasing number of pilot- and industrial-scale applications. In this framework, the possibility of selectively and volumetrically heat compounds of interests in ores or industrial byproducts represents an interesting advantage compared to more conventional heating techniques. Nevertheless, the use of electrical power instead of combusting carbon-based materials can pose problems of costs and availability of proper microwave sources.

This work will focus on the application of microwaves, at 2450 and 5800 MHz to processing of oxides and ores, with the aim to reduce them to industrial-relevant products. Both carbothermic and metallothermic reduction will be addressed, showing the advantages of microwave-assisted processing, especially when combined with other heating techniques. These techniques can also be used proficiently to recycle industrial byproducts, with current poor or no economical value.

Results, including process energy balance and chemical analysis of the obtained metals will be presented, together with the description of the different metal content distribution in reduced particles, when applying microwave heating.

reduction; metallurgy; minerals; recycling

The development of microwave-assisted VOCs removal system

ORAL 27.07.2016 16:45 Session B (Room 305)

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Volatile Organic Compounds (VOCs) have been widely used as ingredients in various industries such as semiconductor, paint chemical and coating process. It is well known that VOCs emissions are dangerous to human health or cause harm to the environment. In particular, some VOCs are strongly regulated by law because they have been classified as a carcinogen. Over the last decades, several technologies have been employed to remove VOCs emitted from industries. For example, activated carbon (AC) tower is commonly used, mainly due to low initial cost. However, AC should be replaced as often as necessary to keep high VOCs removal efficiency. To resolve this problem, recently, zeolite-based rotor concentrator system has been applied to industries like semiconductor and LCD manufacturing process [A][B]. It is divided into 3 zones such as process, desorption and cooling zone, and is constantly rotated. VOCs are removed from the process zone. In the desorption zone, the hot air causes the zeolite pore structure to release the VOCs absorbed, and then VOCs desorbed are completely oxidized to CO_2 and H_2O by thermal oxidizer system. Finally, to cool down the temperature of the rotor, the desorbed part of the rotor is further rotated to the cooling zone. Therefore, the VOCs concentrator can be used for a long time without the replacement of the rotor. However, it requires a lot of heat energy to desorb and oxidize VOCs in both desorption zone and thermal oxidizer system. In the present study, microwave-assisted VOCs removal system has been investigated to improve energy efficiency as well as excellent VOCs removal efficiency. It includes two reactors containing VOCs adsorbents, microwave heating modules and the catalytic oxidizer system. One reactor adsorbs VOCs emitted at atmospheric temperature while the adsorbent in the other reactor is being regenerated by microwave heating. During regeneration, the temperature of the adsorbent is gradually increased to 200℃, resulting in VOCs desorption. VOCs desorbed are completely oxidized CO_2 and H_2O by the catalytic oxidizer system. Regeneration can be accomplished using a time cycle or on demand by measuring the temperature or VOCs remained in the reactor. After the regeneration, the temperature of VOCs adsorbent in the reactor has been reduced to atmospheric temperature by cooling air. Based on the evaluation conducted, microwave-assisted VOCs removal system achieves VOCs removal efficiency of 95%. Especially, it is > 30% energy efficiency compared to zeolite-based rotor concentrator system using conventional heating because the microwave is focused on the adsorbents heating only, not on air heating or reactor around it, meaning that most of the energy is effectively utilized for desorption of the adsorbed VOCs from the adsorbents. Accordingly, microwave-assisted VOCs removal system seems to be particularly promising as a novel technology.

microwave heating; VOCs adsorbent; catalytic oxidation; energy saving

[A] Y.-C. Lin, F.T. Chang, J. Hazard. Mater. 164 (2009) 517–526.
[B] H. Yamauchi, A. Kodama, T. Hirose, H. Okano, K-I. Yamada, Ind. Eng. Chem. Res. 46, (2007) 4316-4322.

Microwave application to annealing of metal thin films

ORAL 27.07.2016 17:00 Session B (Room 305)

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Microwave heating is successfully applied to anneal metal thin films and its multi-layered structures. Observation of thin film microstructures were performed. It has been demonstrated that microwave magnetic field is effective for inducing eddy current and resulted in Joule heating. Optimum Relationship of directions between film normal and microwave magnetic field vector was investigated.

microwave; metal thin film; anneal; magnetic field; microstructure; conductivity

Description of microwave-assisted dewaxing process for artworks

ORAL 27.07.2016 16:45 Session A (Room 302)

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Currently, dewaxing artworks casts with lost-wax casting processes requires the investment of long processing times and big amounts of energy. The traditional process consists of creating a model made of wax, covering it with ceramic shell, letting the shell dry and applying external heat until the wax melts and leaves the ceramic shell cast. Electric or conventional furnaces are used to melt the wax and due to the high-energy cost many artworks models are dewaxed at the same time.

Usually, the obtained results are not optimal because cracks appear due to expansion of the wax during the melting process so pressure can also be used in order to counteract the internal pressure generated by wax.

In this work, authors present a new-patented process for lost-wax dewaxing in artworks casts by using microwave energy. The method consists of applying susceptors to the wax model before covering it with the ceramic shell.

Permittivity measurements of susceptors and employed materials are presented. They show that microwave-heating absorption is much higher in the susceptors than in the wax.

Electromagnetic and temperature simulations are employed in order to understand what happens during this microwave-assisted dewaxing process. The simulations indicate that the susceptor layer heats at a higher rate than the wax. This indicates that the wax next to this susceptor melts while the rest of the wax is cooler. Due to this the wax can later expand and melt without craking the ceramic shell cast.

Experimental tests indicate that the process can be successfully carried out at with a comparable or higher quality than the traditional process with drastic time reductions.

ceramic shell casting; lost-wax dewaxing; microwave-assisted process; susceptor

[A] J. Monzó-Cabrera, et al., Kinetics of combined microwave and hot air drying of leather, J. Soc. Leather Technol. Chemists, vol. 84, no. 1, pp. 38-44, 2000.

Correlation to dipole moment of distillates as molecular mechanism on essential oils separation by microwave assisted distillation

27.07.2016 14:15 e-Poster Session (Show Room)

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Microwave heating as one of the essential oil recovery methods are ultra-efficient technology of time and energy. On the other hand, we are also studying the aim of finding a characteristic different from the efficiency of the microwave process. For distillation of citrus peel by microwave heating, it was already known that the essential oil component of the oxygen-containing compounds are often recovered. That is considered that there is a different separation mechanism from the normal heating. Amount of heat energy by microwave irradiation, the dielectric loss of molecules that receive the microwave irradiation, are considered to be determined by the magnetic loss. However, in the case of mixing of different molecules, thermal energy is not determined by the linear combination of their parameters. Effect of intermolecular interactions formed by the molecule added. Moreover, conduction losses, dielectric loss, magnetic loss, the value changes in response to temperature. In this way, it is very difficult to calculate very complex for the amount of energy. So-called is considered a non-equilibrium process, it can be said that the phenomenon of thermal phenomena and non-thermal phenomena were mixed. Among previous studies, we irradiated with microwaves to made liquid by two compounds, the degree of heating by the mixing ratio of the compound has been found very different phenomena. In the revealing process of the molecular mechanisms from these phenomena,

we thought which be applied to the material separation technology. Therefore, we examined the operation of heating distilling useful substances from citrus and seeds. As a result, by analysis of the extracted components, conventional heating and microwave heating showed a different behavior. Therefore, in this study, focusing on vacuum microwave distillation, to clarify the difference between conventional heating, aimed to reveal their molecular mechanisms. In this study, we took up the process to separate the essential oils from the citrus and grass as a model experiment. It was compared with the conventional method and the vacuum microwave distillation method of steam distillation method. In this study, dielectric loss factor and dielectric constant of the recovered compound, or to compare the physicochemical parameters such as dipole moment. As a result, the molecular mechanism of the distillation revealed. Regardless of the oxygen-containing, we found that could evaluate the value of the dipole moment of the extraction molecule on microwave assisted distillation.

dipole moment; essential oils; microwave assisted distillation; molecular separation mechanism

Impact of microwave drying on total phenolic content and colour of onion slices

27.07.2016 14:15 e-Poster Session (Show Room)

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Onion (*Allium cepa* L.) ranks third in the world production of 7 major vegetables. Onions in dehydrated form have wide demand in the world due to better storage and easy-to-use properties [A]. Dried onion has use as flavour additive in wide variety of food formulations such as soups, sauces, salad dressings and meat products, and traded in the flaked, minced, chopped and powdered forms [B]. It was reported that existing convectional hot air drying methods for the onions cause undesirable changes in colour and nutritive value. Due to this quality defects, researches tend to focus on innovative drying techniques such as microwave assisted drying [B]. Besides reduced loss of quality in the product, microwave drying is advantageous over convective drying for its low drying time and energy consumption.

The aim of this study is to investigate the impact of microwave drying technique on two quality parameters of dehydrated onion slices: total colour change and total phenolic content. For this purpose; 2^3 factorial experimental design with two replicates was used to investigate the effects of salt concentration of pretreatment, power of microwave oven, thickness of slices factors between the levels of 0 – 8%, 80 – 400 W and 3 – 7 mm, respectively. The results were compared to the convectional drying of onion slices part of the study where the effect of salt concentration of pretreatment, temperature, thickness of

slices factors between the levels of 0 – 8%, 50 – 70℃ and 3 – 7 mm, respectively were studied with a two-replicate 2^3 factorial experimental design.

Total phenolic content (mg gallic acid/g dry mass) of the onion slices was analyzed according to [C]. For total colour change (ΔE); *L*, *a*, *b* values of three slices was measured from three different regions by Minolta Chroma meter CR 400 colour meter and their average was used to calculate ΔE according to [B] where unprocessed onion was taken as reference point.

It was observed that maximum obtained total phenolic content of the onion slices for microwave drying (at 0% salt concentration, 400 W and 3 mm slice thickness) were 1,7 times higher than the convectional drying technique (at 0% salt concentration, 70℃ and 3 mm slice thickness) in line with the findings of [D]. It was also twice higher than the total phenolic content of fresh onion due to liberation of phenolics from the food matrix. Total color change was also superior with the microwave drying technique to the convectional one with the ΔE values obtained as 1.2 and 8.8, respectively. These results showed that the investigated factors have significant effects on the total phenolics and total colour change of the onion slices both dried by microwave and convectional technique. However, microwave drying of onion slices gave better quality product with respect to total phenolics and total colour change, by saving time and energy.

microwave drying; onion; quality attributes; factor screening

[A] M.K. Grewal, et al., A less energy intensive process for dehydrating onion, J. Food Sci. Technol., 52, 2, 1131-1137, 2015.
[B] N.R. Sahoo, et al., Impact of pretreatment and drying methods on quality attributes of onion shreds, Food Technol. Biotechnol., 53, 1, 57-65, 2015.
[C] X. Lu et al., Determination of total phenolic content and antioxidant capacity of onion (Allium cepa) and shallot (Allium oschaninii) using infrared spectroscopy, Food Chem., 129, 637-644, 2011.
[D] D. Aslan & M.M. Özcan, Study the effect of sun, oven and microwave drying on quality of onion slices, LWT-Food Sci. Technol., 43, 1121-1127, 2010.

Effects of microwave irradiation on spore-forming bacteria

POSTER 27.07.2016 14:15 e-Poster Session (Show Room)

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As a survival strategy system microorganisms of the genus Bacillus, such as Bacillus natto, to form a very durable spores to the heating and disinfectants. Therefore, effective sterilization techniques have been sought in the medical and food fields. On the other hand, the microwave is one of an electromagnetic wave has been used as a heating device, also continues still debate thermal effect and non-thermal effects (microwave effect). We have

found the microwave heating of the peculiar phenomenon, such as an increase of the growth rate of the bacteria in the microbial cultivation under microwave heating, or the shape change of microorganism by microwave heating. These phenomena can not yet explain. So, we were dealing with spore-forming bacteria as a model microorganism in this study. For each case where phase and spore-forming bacteria that normally cells of spore-forming bacteria form spores to return to normal cells, we compared the conventional heating and microwave heating. The experimental method was as follows. And it was examined the effect of microwave heating is applied to each stage. Further, from the results revealed microwave irradiation effect on the life cycle of the mechanism of spore-forming bacteria. This means the heat stress and electromagnetic stress. We also explored the possibility of sterilization techniques for the spore-forming bacteria. The effect of microwave irradiation at the stage where normal vegetative cells to form spores, were experiments using Bacillus subtilis. After the pre-culture in a nutrient-rich LB medium, it was investigated by comparing the normal culture in the external heating of the microwave culture and the conventional method that uses a spore-forming medium (DSM medium). Microwave cultures using the microwave apparatus of the multi-mode type of frequency 2.45 GHz. Further, the precise control of culture temperature and the microwave output in a jacketed culture tube. Culture temperature was set to aerobic conditions in the optimum temperature of Bacillus subtilis (50℃). It confirmed the growth process of the cells in the cell concentration change and the colony count by absorbance measurement. In addition, in the form observed with an optical microscope by cell staining of malachite green and safranin (Wirtz method). The process of spore returns to the vegetative cells, were studied using the Bacillus subtilis var. Natto of spore state. Germination from spore state amino acids and sugar is to trigger. However, since the temperature condition is also factors work was performed in microwave irradiation.

Bacillus natto; microwave heating; spore-forming bacteria

Performance analyses of mechanically-assisted silent microwave-drills for concrete

27.07.2016 14:15 e-Poster Session (Show Room)

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The microwave-drill concept employs the locally induced thermal-runway instability as a means to create soften hotspots in order to alleviate the debris removal from the drilled hole [A]. A prototype of a silent, remotely-operated microwave drill [B] has demonstrated a capability of drilling 12 mm diameter holes of 26 cm depth in concrete. Here we present experimental analyses of this microwave-drill performance in various configurations. A simplified numerical model of the microwave-drilling operation provides a benchmark for these analyses. Several means for the removal of the molten and softened concrete were applied in these experiments, including air pressure and other mechanical tools. The

drilling speed and the other operating parameters were examined in various conditions. The highest drilling speed achieved so far is 2.8 cm/min. The minimal speed required for practical needs is considered as 5 cm/min, which is yet a goal to be achieved in this study.

microwave heating; hotspot; concrete; drilling

[A] E. Jerby, V. Dikhtyar, O. Actushev and U. Grosglick, The microwave drill, Science, Vol. 298, pp. 587-589, 2002.

[B] E. Jerby, Y. Shamir, R. Peleg and Y. Aharoni, A silent mechanically-assisted microwave drill for concrete with integrated adaptive impedance matching, Proc. AMPERE-2013, 14th Int'l Conf. Microwave & High-Frequency Heating, pp. 267-270, Nottingham, UK, Sept. 2013.

Incremental solidification (toward 3D-Printing) of metal powders by localized microwaves

27.07.2016 14:15 e-Poster Session (Show Room)

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This paper presents several experiments and theoretical analyses oriented to study the feasibility of various localized microwave-heating (LMH) schemes for 3D-printing and additive-manufacturing processes. Following Refs. [A][B], the incremental solidification by LMH of ~1 mm^3 batches of various metal powders is tested in various conditions, and compared to the commonly used laser-based process. Alternative approaches proposed, of using the raw material in forms of solid or compact-powder wires, are examined as well. A theoretical LMH model is employed in order to evaluate the different mechanisms of the microwave-matter interaction (e.g. magnetic heating due to eddy current, micro-plasma breakdown, etc.) dominating the various LMH schemes. SEM, XRD and hardness analyses of the LMH solidified products are examined in the various cases. Practical considerations, including the implementation of transistor-based (LDMOS) LMH generators [C] in compact schemes, are presented. The LMH applicability for 3D-printing and additive-manufacturing processes is discussed in view of the presently available techniques.

additive manufacturing; 3D printing; localized microwave heating; sintering

[A] E. Jerby, X. Planta, R. Rubio, A. Salzberg, B. Cavallini, Y. Meir, Method and devices for solid structure formation by localized microwaves, US Patent App. 20140021171 A1 (also published as EP2689635A1 and WO2012127456A1).

[B] E. Jerby, Y. Meir, A. Salzberg, E. Aharoni, A. Levy, J. Planta Torralba and B. Cavallini, Incremental metal-powder solidification by localized microwave-heating and its potential for additive manufacturing, Additive Manufacturing, vol. 6, pp. 53-66, 2015.

[C] Y. Meir and E. Jerby, Localized rapid heating by low-power solid-state microwave-drill, IEEE Trans. Microwave Theory & Tech., vol. 60, pp. 2665-2672, 2012

Topic 07: Material synthesis

Localized microwave-heating in solids, plasma and powders, and its potential (e.g. for 3D-printing)

26.07.2016 09:40 Main Room

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The paper reviews in a paradigmatic approach the effects of localized microwave-heating (LMH), hotspot formation and intentionally-induced thermal-runaway instability [A] (in solids and in metal powders), as well as various applications derived from the microwave-drill technique [B]. These include for instance local heating up to >1000K using LDMOS transistors [C], thermite-powder ignition in air atmosphere and underwater [D], generation of plasma columns and spheres by LMH from molten hotspots (e.g. silicon [E] or titanium [F]), production of dusty-plasma and nano-powders; and local solidification of metal powders and 3D-printing experiments [G]. The potentials and limitations of LMH devices, e.g. as low-cost substitutes for lasers, and in particular the feasibility of LMH applications in various 3D-printing and additive-manufacturing processes are discussed.

microwave heating; dusty plasma; nano-powders: additive manufacturing

[A] E. Jerby, O. Aktushev and V. Dikhtyar, Theoretical analysis of the microwave-drill near-field localized heating effect, J. Appl. Phys., Vol. 97, 034909, 2005 (and references therein).
[B] E. Jerby, V. Dikhtyar, O. Actushev and U. Grosglick, The microwave drill, Science, Vol. 298, pp. 587-589, 2002.
[C] Y. Meir and E. Jerby, Localized rapid heating by low-power solid-state microwave-drill, IEEE Trans. Microwave Theory & Tech., vol. 60, pp. 2665-2672, 2012.
[D] Y. Meir and E. Jerby, Underwater microwave ignition of hydrophobic thermite powder enabled by the bubble-marble effect, Appl. Phys. Lett., vol. 107, 054101, 2015.
[E] Y. Meir, E. Jerby, Z. Barkay, D. Ashkenazi, J. B. A. Mitchell, T. Narayanan, N. Eliaz, J-L LeGarrec, M. Sztucki and O. Meshcheryakov, Observations of ball-lightning-like plasmoids ejected from silicon by localized microwaves, Materials, vol. 6, No. 9, pp. 4011-4030, 2013.
[F] S. Popescu, E. Jerby, Y. Meir, Z. Barkay, D. Ashkenazi, J. B. A. Mitchell, J-L. Le Garrec and T. Narayanan, Plasma column and nano-powder generation from solid titanium by localized microwaves in air, J. Appl. Physics, vol. 118, 023302, 2015.
[G] E. Jerby, Y. Meir, A. Salzberg, E. Aharoni, A. Levy, J. Planta Torralba and B. Cavallini, Incremental metal-powder solidification by localized microwave-heating and its potential for additive manufacturing, Additive Manufacturing, vol. 6, pp. 53-66, 2015.

Hydrogen from solids by microwave influence

ORAL 26.07.2016 11:15 Session B (Room 305)

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The increasing share of solar and wind in the energetic mix and the COP21 agreement pave the way for a new family of technologies based on renewable energy. Storage of this energy in form of hydrogen is one of the most explored challenges in order to reach a sustainable society. The challenge is being addressed using storage systems based on compressed, liquefied and materials-bounded hydrogen. Hydrides can store high amounts of hydrogen but this usually comes at the expense of higher desorption temperatures and consequently the efficiency will suffer as a result of the heat losses of the system. What if we find a way to deliver energy directly to the material? In fact, we do this every day at home... Just use microwaves!

Microwaves are used in this works to directly heat the material to control the hydrogen release opening the possibility of dealing efficiently with high capacity hydrides. Moreover, we have designed a material sensitive to microwaves and at the same time with high porosity to store more hydrogen in small spaces. This technology has been subject of a national patent application [A].

H_2 storage; hydrides; microwave heating; aerogels; supercritical CO_2

[A] L. M. Sanz, M. Rueda, A. Navarrete and Á. Martín. Patente Nacional 201500170

Microwave sintering of nanoporous ceramic membranes modified with carbon nanotubes

ORAL 26.07.2016 11:30 Session B (Room 305)

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Membrane technologies are popular in various industries. Porous ceramic membranes have numerous merits, among which are high heat resistance (such membranes are serviceable until 1000℃), chemical inertness, and high mechanical strength. Fine-pored ceramic

membranes are widely used in the purification of gases and liquids, including drinking and waste water. There are several methods to control the porous structure of ceramic membranes, the embedding some additives such as carbon nanomaterials being particularly promising. The application of MW heating for the sintering the ceramic materials has certain specific features: a significant (50℃–100℃) decrease in the process temperature, especially at the intermediate stage of sintering; and a shortening in the high-temperature stage of the sintering process due to rapid bulk heating. In some cases, MW sintering like conventional sintering allows the production of ceramics with the homogenous particle size distribution, while the obtained pore size distribution can be even more homogenous [A]. It is supposed, that integration of both these methods can result in sintering the ceramic membranes with advanced porous structure.

This paper presents the results of the microwave sintering of nanoporous ceramic membranes containing carbon nanotubes (CNTs).

The samples (80 wt.% Al_2O_3 + 17 wt.% (CNT - SiO_2) + 3 wt.% La_2O_3) shaped as a disk (diameter 30 mm, height 3 mm) were annealed under anaerobic conditions at temperature T = 950℃ during 1 hour under conventional and MW heating. The MW sintering was carried out using a lab scale set up (power up to 1 kW) based on a cylindrical single-mode cavity, which is excited by oscillation mode H_{011}. The sintering and transformation of initial oxide phases were studied. It was found that the MW sintering results in a stronger transformation of initial oxide phases and agglomeration of the sample. The experimental data have demonstrated the noticeable increase of the amount of high-temperature aluminum oxide (δ-Al_2O_3) and decrease of the total pore volume due to agglomeration of micropores and small mesopores after the MW sintering. Hence, water permeability of the MW sintering membrane was greater due to the increase of the amount of large mesopores (>15 nm).

The present research demonstrates the capability of MW sintering of ceramic membranes with advanced porous structure.

microwave sintering; ceramic membranes; carbon nanotubes

[A] K. I. Rybakov, et al., Microwave Sintering: Fundamentals and Modeling, J. Am. Ceram. Soc., vol. 96, no. 4, pp. 1003-1020, 2013.

Microwave direct flash synthesis of the $SrFe_{12}O_{19}$ hexaferrite

ORAL 26.07.2016 11:45 Session B (Room 305)

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Hexaferrites compounds are used worldwide at large scale as permanent magnets. In that respect, many studies have been carried out to reduce their production cost, using usually the conventional solid state route. Accordingly, one research strategy consists in decreasing the temperature and duration time of the reaction, in working on the powder reactivity.

Another strategy consists in innovative synthetic routes such as the microwave technique processes [A]. The latters are often reported to decrease both the temperature and the time needed to complete the synthesis. Among the previous studies already published, one can cite the liquid route synthesis of hexaferrites, using microwave hydrothermal method or the microwave combustion method. Only two experiments related to reactive sintering of hexaferrites have been done so far. In this study, we focus on the synthesis of the M-type strontium hexaferrite $SrFe_{12}O_{19}$ in a 2.45 GHz single mode microwave cavity [B]. Starting from cold pressed samples, $SrCO_3$ and Fe_2O_3 mixtures (1:6 ratio) have been heated up in a microwave cavity, in either electric or magnetic fields. The heating behavior is discussed accordingly the different experimental conditions, as well as the final properties of the products (microstructure and magnetic properties). It is clearly shown that single-phase hexaferrites materials can be produced in less than 30 min, for our optimal experiment conditions [C]. Their properties are compared with those of ceramics obtained by conventional solid state reaction. microwave sintering, ceramic membranes, carbon nanotubes.

material synthesis

[A] D. K. Agrawal, Microwave processing of ceramics, Curr. Opin. Solid State Mater. Sci., vol. 3, pp. 480-485, 1998.

[B] A. Thuault, E. Savary, J.-C. Hornez, G. Moreau, M. Descamps, S. Marinel, and A. Leriche, Improvement of the hydroxyapatite mechanical properties by direct microwave sintering in single mode cavity, J. Eur. Ceram. Soc., vol. 34, pp. 1865-1871, 2014.

[C] F. Molinari, E. Savary, A. Maignan, S. Marinel, Microwave direct flash synthesis of the SrFe12O19 hexaferrite, *paper to be published*

Effects of microwave sintering on the microstructure and magnetoelectric properties of PZT/$CoFe_2O_4$ composites

26.07.2016 12:00 Session B (Room 305)

C. P. F. Perdomo [A], F. L. Zabotto [B], D. Garcia [B], R. H. G. A. Kiminami [A]
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This paper compares the effects of microwave-assisted versus conventional sintering on the microstructural and physical characteristics of nanocrystalline (x)PMN-PT/(1-x)Fe_2CoO_4, where x = 0.5, 0.6, 0.7 and 0.8, prepared in situ via the Pechini method, to obtain a biphasic system with a highly homogeneous two-phase distribution. The sintered samples were microstructurally characterized by scanning electron microscopy (SEM) and X-ray diffraction (XRD) and their apparent density and porosity were measured by the Archimedes method. These analyses indicated that the sintering method yielded diphase ceramics with homogeneous microstructures, excellent distribution of the ferromagnetic phase (Fe_2CoO_4) in the ferroelectric matrix, and high electrical resistivity. In addition, the magnetoelectric response of the composites indicated that the sintering process had a significant effect on their grain size and magnetoelectric properties.

synthesis; microwave sintering; PZT-$CoFe_2O_4$; properties

Microwave processing of solid-state electrolyte for Li-ion batteries

26.07.2016 16:00 Session B (Room 305)

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$Li_{1+X}Al_XGe_{2-X}(PO_4)_3$ (LAGP) glass system is considered as a promising solid state electrolyte for Li-ion batteries. The crystallization of LAGP glass was investigated using 30 GHz high frequency microwave processing. The LAGP glass was prepared via the regular melt-quench method. Thermal, chemical analyses and X-ray diffraction (XRD) were performed to characterize the prepared glass and to design the heat-treatment regime. The ionic conductivity of the microwave treated glass was measured using impedance spectroscopy. The microwave treated glass was also characterized using XRD and scanning electron microscopy (SEM). High frequency microwave heating was successfully used to crystallize LAGP glasses indicating that this is a promising candidate technology. The ionic

conductivity of the microwave crystallized LAGP samples was relatively higher than the corresponding LAGP conventionally crystallized samples at the same conditions.
This project is funded by the Helmholtz Association in the Helmholtz Energy Alliance "Stationary Electrochemical Solid State Storages and Converters" under the grant HA-E-0002.

microwaves; solid-state electrolyte; LAGP; crystallization

Microwave synthesis of high entropy alloys comprising at least one ferromagnetic element

26.07.2016 16:15 Session B (Room 305)

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High entropy alloys (HEA) are a class of multi-component alloys composed of five or more principal constituent elements, none of which is predominant, and each with a concentration between 5 and 35 atomic % [A]. These alloys have a tendency to form simple structures, like face centered cubic (FCC) and body centered cubic (BCC), instead of intermetallic compounds. Several production techniques have been tried to synthesize these materials [B]. According to the literature research, up to now, most of the production attempts proceeded from the liquid state (arc melting, induction melting), from the solid state (mechanical alloying, powder metallurgy), from the gas state (sputtering techniques, mainly for coatings) and from electrochemical processes. In this work a new microwave-assisted near-net-shape technology, using the powder metallurgy approach for the preparation of HEA, will be investigated, showing how it is able to overcome the limits of current melting technologies, which tend to originate defects in the cast products, like voids, porosity and segregations.

In this work, different HEA have been prepared, comprised of at least one ferromagnetic element and one highly reactive element couple, like Al-Ti, Al-Ni and Al-Fe, in order to improve heat generation due to both the magnetic field contribution (microwave heating) and the exothermal contribution occurring during synthesis. Ceramic-particles reinforced high entropy alloys will be presented as well, together with the use of recycled metals deriving from machining operations of lighweight alloys (Al, Ti).

reduction; metallurgy; minerals; recycling

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[B] Zhang, Y.; Zuo, T.T.; Tang, Z.; Gao, M.C.; Dahmen, K.A.; Liaw, P.K.; Lu, Z.P. Microstructures and properties of high-entropy alloys. Prog. Mater. Sci. 2014, 61, 1–93.

Flash microwave sintering of ceramics

ORAL 26.07.2016 16:30 Session B (Room 305)

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We present results of a study of ultra-rapid (flash) sintering of ceramics under 24 GHz microwave heating with high power density absorbed in the material (10 – 500 W/cm3). Oxide ceramic samples of various compositions – Al_2O_3, $Al_2O_3(Y_2O_3)$, Y_2O_3, $MgAl_2O_4$, Yb:$(LaY)_2O_3$ – were sintered to a density above 0.98 – 0.99 of the theoretical value in 0.5 – 5 minutes without isothermal hold. An analysis of the experimental data, including the microwave power evolution during the process, heating and cooling rates, and microstructure characterization (SEM, XRD), provides insight into the mechanism of flash sintering [A].

Flash sintering occurs when the temperature and microwave radiation intensity satisfy the conditions of the development of thermal runaway that arises due to sharp increase of the effective conductivity of the material with temperature. The defects of the crystalline structure and the presence of impurities lead to preferential absorption of microwave power in the boundary regions of the grains, causing their softening / pre-melting. The enhanced densification in the material with low viscosity of the boundary phase occurs due to rotation and sliding of the grains, which also adjust their shape due to fast mass transport via the (quasi-) liquid phase.

We believe that the same mechanism is relevant for the flash sintering of various oxide ceramics under a DC / low-frequency AC voltage applied to the sample [B]. In this case, volumetric Joule heating caused by a current flowing through the sample triggers, at a certain level of power dissipated per unit volume, a runaway instability that leads to the softening / pre-melting of grain boundaries.

microwave; oxide ceramics; flash sintering; runaway

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[B] M. Cologna, A.L.G. Prette, R. Raj, Flash-sintering of cubic yttria-stabilized zirconia at 750°C for possible use in SOFC manufacturing, J. Am. Ceram. Soc., vol. 94, pp. 316–319, 2011.

Effect of microwave mechanisms to fabricate efficient bioceramic components

26.07.2016 16:45 Session B (Room 305)

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In the last decades, advanced ceramic materials have become widely applied in different technological fields due to their outstanding performance while in service as structural and functional elements. Particularly, in the field of biomaterials engineering, bioceramics have been developed as an alternative to traditionally applied materials, such as metals and polymers, in order to provide the necessary characteristics to replace biological structures within the human body. This relatively new group of ceramic materials adequately meets several requirements that are crucial for biological applications in terms of biocompatibility. One of the most important applications of bioceramics is found in restorative dentistry as prostheses and implants [A]. In this regard, the material must cover certain criteria in terms of mechanical, aesthetic and chemical properties. Some of the most common bioceramics are alumina, zirconia, tricalcium phosphate and hydroxyapatite.

The novel and innovative technologies aim at helping industrial sectors lower their production costs and, at the same time, lessen their environmental impact by cutting down the amount of greenhouse gas (GHG) emissions released into the atmosphere, which also serves to comply with European Union directives that have planned to reduce GHG emissions by at least 20% compared to 1990 levels by 2020 [B].

Particularly, due to the intrinsic dielectric properties of zirconia, microwave sintering represents an interesting opportunity at consolidating zirconia powders with a reduced processing time and energy consumption by utilizing electromagnetic radiation to provide high-enough temperatures that allow full densification of the material. The most important advantages of microwave sintering against conventional sintering methods are [C][D]: Shorter sintering time and lower energy consumption, fast heating rates, materials with a finer (nanometric) microstructure with a high degree of densification and enhanced mechanical properties may be obtained due to the densification mechanisms involved and flexible due to the possibility of processing near-net-shape materials.

microwave sintering; bioceramics; mechanical properties; hydrothermal behavior

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[B] EUROPE 2020: A strategy for smart, sustainable and inclusive growth. Brussels: European Commission, 2010.
[C] M. Oghbaei, et al., Microwave versus conventional sintering: A review of fundamentals, advantages and applications. J. Alloys Compd., vol. 494, pp. 175–189, 2010.
[D] A. Upadhyaya, et al., Microwave sintering of W–Ni–Fe alloy. Scr. Mater., vol. 56, pp. 5–8, 2007.

Microwave synthesis of iridium complexes for OLED and their precise analysis by using a LC-TOF MASS method

26.07.2016 17:00 Session B (Room 305)

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A series of Ir(R-ppy)$_3$ complexes is a well-known phosphorescent emitter for highly efficient organic light-emitting devices (OLEDs). We synthesized Ir(R-ppy)$_3$ complex by using various microwave reactors, and analyzed their dielectric properties and their purity with LC-MS method to find the optimal conditions for the microwave synthesis. New coaxial cable type flow microwave reactor with solid state microwave generator was developed to conduct advanced microwave synthetic process. A OLED fabricated with highly pure microwave synthesized Ir(ppy)$_3$ showed a high power efficiency of over 100 lm/W.

microwave synthesis, Ir(R-ppy)$_3$ complexes, LS-MS, flow microwave reactor

Microwave treatments of MSWI bottom ash

26.07.2016 14:15 e-Poster Session (Show Room)

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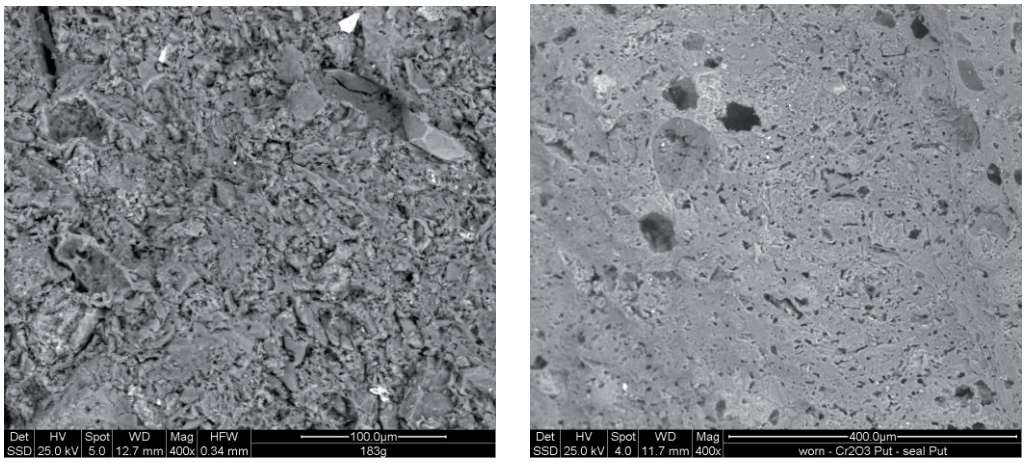
The bottom ashes deriving from the incineration of municipal solid waste-MSWI can be considered as new raw material for ceramic and building sector. Some valorization treatment requires high temperatures to transform them in novel and attractive materials: glass-ceramics, ceramics and glasses [A]. Preliminary results obtained by an innovative microwave-assisted sintering process are reported in this paper.

Microwave heating technique was accurately compared with a conventional thermal treatment with the aim to study the influence of the heat treatment parameters on the crystallization behavior and on the microstructure of the obtained bricks. Particular attention was paid to define the crystallization evolution in the MW field with temperature, where some new crystalline phases were observed [B]. It is shown that together with residual quartz and newly formed anorthite, which are the main crystal phases in the conventionally prepared samples, in some of the ceramics obtained by microwave heat-treatment the precipitation of new sodium rich crystals is observed. In addition, higher crystallinity and lower size of the anorthite crystals phases were observed, which improves the mechanical properties.

The possibility to obtain new bricks, with higher waste concentration with respect conventional thermal treatments, in a very short thermal cycle at relatively low temperatures was demonstrated. Moreover, on the basis of the final properties obtained, microwave sintering of bottom ash is recommended in the manufacture of ceramic products, since it allows reducing the amounts of MSWI ash that would be otherwise disposed in landfills. Furthermore, significant reductions in the consumption of raw materials as well as in the energy demand should be highlighted as positive outcome of the proposed process.



◀ SEM observation on fresh fracture of sintered materials in (left) conventional and (right) MW furnaces. In case of MW treatment, a vitrified ceramics with more adherent grains have been obtained.

incineration bottom ash; sintering; microstructure; fast heating effects

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Microwave heating applied to polymer science

26.07.2016 14:15 e-Poster Session (Show Room)

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During the last 25 years microwave radiation has come to be accepted as a heating source for chemical synthesis due to the number of advantages it offers. These include a reduction in synthesis time, the possibility that it can be used without the need of solvents and, above all, because it is an easy, fast and safe method. Moreover, microwave technology has been shown to be scalable as it is already widely used in the pharmaceutical industry, as a means of synthesizing compounds and polymers, and vulcanized rubber is a noteworthy example.

In our research group, microwave heating has been applied in polymer science since years. In fact, it has been used not only as a drying technique, after the synthesis reactions [A], but also as the heating method for the whole process of polymerization. The use of microwave heating results in a very efficient technique for the synthesis of polymers, and more specifically for one type of polymer named resorcinol - formaldehyde (RF) xerogels, obtained by sol - gel methodology. The synthesis of RF xerogels induced by microwaves presents many advantages such as a very rapid process (i.e., 4 - 5 hours versus 4 - 5 days in a conventional synthesis), a one-pot synthesis, and the possibility of a total control the processes occurring. It is even possible to know which step of the process (heating, polymerization, curing, drying) is occurring [B], in order to stop the process in a specific step to control the final form of the product [C]. Different aspects of the microwave synthesis have been studied, such as residence time, temperature or specific power used, and their influence on the final properties of the RF xerogels have been evaluated [D]. These parameters have been used for scaling up the process [E] and currently our spin - off is producing this type of polymers at an industrial scale (www.xerolutions.com). Therefore, a completely controlled and efficient process of polymerization is obtained by means of microwave heating and all the chemical variables involved can be studied [F][G] for obtaining bespoke materials.

microwave; oxide ceramics; flash sintering; runaway

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[G] I.D. Alonso-Buenaposada, N. Rey-Raap, E.G. Calvo, J.A. Menéndez, A. Arenillas, Effect of methanol content in commercial formaldehyde solutions on the porosity of RF carbon xerogels, Journal of Non-Crystalline Solids, 426, pp. 13 - 18, 2015.

Improved microwave-assisted synthesis of rare earth phthalocyanines

26.07.2016 14:15 e-Poster Session (Show Room)

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The phthalocyanines are organic macrocycle with extended π system, that accounts for its strong absorption around 670nm. In their cavity, more than 70 metal ions have been hosted and among them the rare earth have attracted the attention for their ability to coordinate two rings per each ion (the so called 'sandwich').

In the recent past phthalocyanines used as sensitizer of TiO_2 knew good reputation for enhancing the photoactivity of the semiconductor, extending the absorption of TiO_2 in the visible range and charges lifetime.

In 2007 Mele et al [A] worked on rare earth phthalocyanines used as sensitizers showing very promising results. In this view the synthesis of rare earth phthalocyanine, namely Gd, Sm, Yb, Er and Nd ones, is a great challenge for the reaction time and intrinsic low yield. Currently, a synthesis last from 3 to 24 hours depending to the metal with yield from 18 to 25%

A microwave-assisted solvent-free synthesis of lipophilic phthalocyanine is therefore proposed in the present work in order to enhance the yield while concurrently lowering the reaction time. Indeed, preliminary results obtained on unsubstituted $Gd(Pc)_2$ showed 30% of yield in 1hr of synthesis performed in a microwave single mode applicator operating at the frequency of 2.45 GHz and with an output power level ranged between 30

and 300 W. The same promising approach has been extended to other rare earth phthalocyanine, and the experimental results are fully detailed in this work.

To our knowledge it is the first time that rare earths phthalocyanines have been synthetized by microwave assisted reaction, contrary to d-metal ones [B].

phthalocyanine; rare earth cations; organic synthesis; solvent-free; single mode applicator

[A] G. Mele, E. Garcia-Lopez, L. Palmisano, G. Dyrda, and R. Slota, "Photocatalytic Degradation of 4-Nitrophenol in Aqueous Suspension by Using Polycrystalline TiO2 Impregnated with Lanthanide Double-Decker Phthalocyanine Complexes," Journal of Physical Chemistry C, vol. 111, no. 17, pp. 6581–6588, May 2007.
[B] Shaabani, R. Maleki-Moghaddam, A. Maleki, and A. H. Rezayan, "Microwave assisted synthesis of metal-free phthalocyanine and metallophthalocyanines," Dyes and Pigments, vol. 74, no. 2, pp. 279–282, Jan. 2007.

Two-step microwave sintering of nanostructured ZnO-based varistor

POSTER 26.07.2016 14:15 e-Poster Session (Show Room)

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Fine microstructures have been generating considerable interest in the search for more efficient varistors, due the enhanced electrical properties that can be achieved with homogeneous fine-grained microstructures. Although the desired microstructure involves enhanced properties, controlling grain growth is still a problem when very fine particles, such as nanoparticles, are used as starting materials. Fast processing techniques such as microwave sintering have been developed to control grain growth, but the resulting microstructures are still quite coarse. The two-step sintering technique is a promising method for preserving finer microstructures, since it restrains unstable microstructures during densification, controlling grain growth. However, this technique requires long holding times at low temperatures. The combination of the successful two-step sintering method with fast microwave sintering, however, is a promising technique to obtain fine microstructures with enhanced electrical properties. In this work, samples of nanostructured ZnO-based varistor compositions doped with 1%-mol of Sb_2O_3 and 0.5%-mol of bismuth, cobalt, manganese and chrome oxides were processed by the two-step microwave sintering technique. The samples were heat-treated at 1050, 1100 and 1150 ℃ for 1 min, applying heating rates of 100℃/min, then cooled at 50℃/min to 850 and 900℃, at which temperature they were held for 10, 30 and 60 min. The microstructure, density and electrical properties were analyzed. Dense samples with 96% of theoretical density and an average grain size smaller than 2.1 μm were obtained. The samples exhibited enhanced electrical properties, such as non-linearity coefficient and breakdown electrical field higher than 30 and 9 kV/cm, respectively, and leakage current below 70 μA.

Microwave assisted combustion synthesis of $AlFe_2B_2$ for magnetic refrigeration

26.07.2016 14:15 e-Poster Session (Show Room)

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Magnetocaloric materials are a new class of multifunctional materials appropriate for thermal management applications (e.g. magnetic refrigeration) that exhibit significant "caloric" effects: they experience temperature changes under isothermal or adiabatic variations of a magnetic field [1]. Many of these materials contain rare, toxic or strategically-limited elements: these materials include Gd, $Gd_5(GeSi)_4$, FeRh, MnAs, FeMnAsP and$(La((FeCo)Si(H))_{13}$ [A].

A promising system is $AlFe_2B_2$, lightweight and composed of earth-abundant, easy recyclable and safe elements [B]. It has a simple two-dimensional layered structure with a magnetic transition near room temperature, the order of which is presently under debate [A].

Arc furnace melting (in vacuum or in Argon atmosphere) is the most used synthesis technique, but with a strong tendency to segregation and to produce byproducts. Hence, multiple re-meltings, longtime/high temperature annealings and HCl etching are required [A][B], in order to obtain only $AlFe_2B_2$.

In this work we discover a new more simple and low-cost route to synthesize $AlFe_2B_2$ through Microwave Assisted Combustion Synthesis (MACS/MASHS).

The aim of this study is to understand what are the most suitable parameters to obtain a bulk sample in which the reaction yield which is maximized, namely, maximize the amount of $AlFe_2B_2$ phase, minimizing the byproducts (such as different types of Fe-aluminides, and Al/Fe-borides).

Samples were made using a single mode MW applicator (power: 300 mW). We synthesized specimens with different application field mode (E or H), cold forming pressure, weights and with or without SiC susceptors (within mixed with elemental metal powders or outside). The samples were studied by XRD, SEM, Magnetic DSC and Thermal DSC/TGA.

The results show an high percentage of magneto-caloric phase already after MACS synthesis and a magneto-caloric effect with a similar magnitude to the state of the art (specimens mainly produced via arc melting, several re-melting, annealing and HCl etching).

Finally, the MACS route is able to achieve quickly and easily the magneto-caloric phase $AlFe_2B_2$.

ternary compound; magnetocaloric effect (MCE); combustion synthesis; self-propagating high temperature synthesis; magnetic refrigeration

[A] X. Tan, et al., Magnetocaloric Effect in AlFe2B2: Toward Magnetic Refrigerants from Earth-Abundant Elements, J. Am. Chem. Soc., vol. 135, 9553-9557, 2013.
[B] L.H. Lewis, et al., Developing magnetofunctionality: Coupled structural and magnetic phase transition in $AlFe_2B_2$, J. All. Comp., vol. 650, pp. 482-488, 2015.

Topic 08: Microwave assisted extraction

Methane dry reforming by microwave heating

ORAL 27.07.2016 18:00 Session A (Room 302)

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Microwave-activated gas-solid reactions have attracted increasing attention over the last 20 years, mostly due to the ability of microwaves to selectively interact with metal nanoparticles [A]. Perovskite structured catalysts are known for their good thermal and structural stability as well as for their high oxygen storage capacity at high temperatures [B]. Perovskites have also gained considerable attention for synthesis and catalytic applications under microwaves.

Our goal is to find solutions for some major challenges in microwave-assisted processes, such as (1) accurate temperature measurement, (2) homogeneous heat distribution in a microwave field and (3) investigation and impact of hot spot generation on the perovskite catalytic performance under the application of a microwave field for high temperature gas-solid reforming reactions [C].

A novel microwave reactor with adaptable geometry allowing for uniform electric field distribution over the volume of the reactor and wall temperature monitoring by means of a thermal camera has been developed. Synthesis, characterization and dielectric properties measurement of perovskite catalysts have been performed. Experimental results from the microwave-assisted methane dry reforming processes will be presented. The microwave-assisted process will be compared with the conventionally heated counterpart in terms of product yield, selectivity and energy efficiency.

microwave; perovskite; reforming; process intensification

[A] Prado-Gonjal J, Arévalo-López ÁM, Morán E. Microwave-assisted synthesis: A fast and efficient route to produce LaMO3 (M=Al, Cr, Mn, Fe, Co) perovskite materials. Mater Res Bull. 2011;46(2):222-230. doi:10.1016/j.materresbull.2010.11.010.

[B] Navarro RM, Peña M a, Fierro JLG. Hydrogen production reactions from carbon feedstocks: fossil fuels and biomass. Chem Rev. 2007;107(10):3952-91. doi:10.1021/cr0501994.

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Yield vs selectivity in grape pomace polyphenol microwave extraction

ORAL 27.07.2016 18:15 Session A (Room 302)

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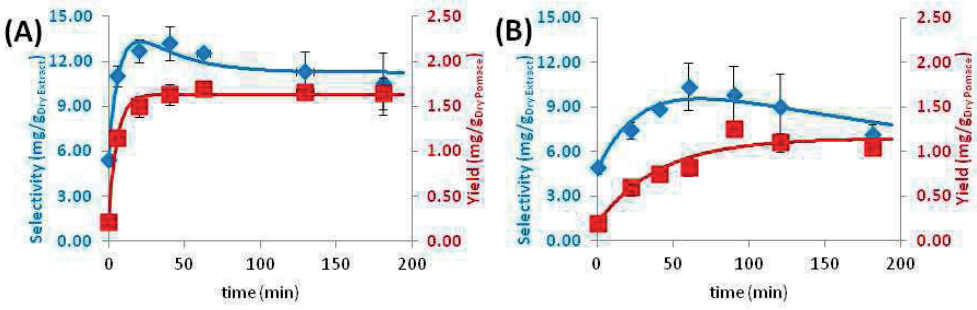
[B] Instituto de Biología Experimental Tecnológica, Instituto de Tecnología Química e Biológica, António Xavier, Universidade Nova de Lisboa, Avenida da República, Quinta do Marquês, 2780-157 Oeiras, Portugal.

The advantages of microwave extraction against conventional processes have been already widely discussed. However, nothing has been said about extraction selectivity. In this work, short microwave-pressure pretreatments to the conventional process have been assessed. The objective is to improve both yield and polyphenol selectivity. Microwave radiation greatly accelerates polyphenol extraction, while other compounds (mainly sugars and fibres) do not present such a considerable improvement. Thus, microwaves affect differently to each type of compounds. By controlling the kinetics of the extraction, it is possible to increase yield a 57%, enhance selectivity a 32% and reduce extraction time to a fifth. Shorter extraction times avoid the massive extraction of undesired compounds and so, the polyphenol richness decay is prevented. Microwave-pressure pretreatments allow to stop the extraction at an optimal time where both maximum yield and selectivity can be achieved; whereas this is not possible in conventional extraction. In the case of anthocyanins (group of special interest due to their antioxidant properties), the microwave boost is even more pronounced, as it is possible to obtain a final product 85% richer in these valuable compounds. This improvement is shown in Figure A.



◀Anthocyanin selectivity and yield kinetics for (A) microwave pretreatment of 300 W during 60 s and (B) conventional solid-liquid extraction at 60℃.

Regarding biofunctionality, microwave extract compositions differ from conventional extracts, and so do their antioxidant activity. In vivo assays provide different antioxidant capacities than in vitro results. The latter are almost proportional to the polyphenol concentration, whereas cellular antioxidant analyses show that microwave extracts are able to preserve their antioxidant properties for longer than the conventional does. Therefore, an interaction between polyphenols and other substances that affect their bioavailability is likely.

These results suggest the convenience of implementing this kind of technology in order to improve product quality and assure the sustainability of winery industry by exploiting a value-less residue.

microwave pretreatment; polyphenols; yield; selectivity; antioxidant activity

Microwave-assisted extraction of phytochemicals

27.07.2016 18:30 Session A (Room 302)

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Several epidemiological studies highlighted the importance of a diet based on fruits and vegetables, for a significant reduction in the risk of several human health diseases [A]. Therefore, the extraction of the so-called dietary phytochemicals started gaining an extraordinary research interest from both academia and industries, to the detriment of their synthesis, particularly when the concepts and principles of Green Extraction [B] and Process Intensification [C] are considered, in order to pursue a sustainable development.

Among the green extraction techniques, the application of microwave energy is surely to be considered one of the most appealing ones, mainly as a direct consequence of its unique heating mechanism. According to the nature of the plant matrix as well as of the phytochemical compounds of interest, microwaves can be applied in a plenty of variants.

Aim of the present work is to display some of our recent results obtained by microwave assisted extraction (MAE) of phenolic compounds from Juglans regia L., Cinnamomum Zeylanicum and Curcuma longa L. [D], highlighting the advantages, when any, over more conventional extraction procedures as well as over the chemical synthesis of those molecules, also in terms of energy consumptions as well as environmental assessments of these different approaches.

Phytochemicals; green extraction; microwaves; environmental assessment

[A] Y.J. Surh, Cancer chemoprevention with dietary phytochemicals, Nature Rev. Cancer vol. 3, pp. 768-780, 2003.
[B] F. Chemat, M.A. Vian, G. Cravotto, Green extraction of natural products: concept and principles, Int. J. Mol. Sci. vol. 13, pp. 8615-8627, 2012.
[C] A. Stankiewicz, J.A. Moulijn, Process Intensification: transforming chemical engineering, Chem. Eng. Prog. vol. 96, pp. 22-34, 2000.
[D] E. Zerazion, R. Rosa, E. Ferrari, P. Veronesi, C. Leonelli, M. Saladini, A.M. Ferrari, Phytochemical compounds or their synthetic counterparts? A detailed comparison of the quantitative environmental assessment for the synthesis and extraction of curcumin, Green Chem. (2016), DOI:10.1039/c6gc00090h.

Simultaneous microwave & ultrasound–assisted process for synthesis of functionalized metal nanoparticles used for control of mosquito vectors

ORAL 27.07.2016 18:45 Session A (Room 302)

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Preventing or reducing the transmission of dengue virus depends either on controlling the mosquito vectors or on the interruption of human–vector contact. WHO promotes the strategic approach known as Integrated Vector Management (IVM) to control mosquito vectors, including those of dengue [A].

Present paper describes the simultaneous microwave & ultrasound – assisted process for the synthesis of functionalized copper nanoparticles using different essential oils as the raw material for the repelling agent used for mosquito vectors control.

Different types of essential oils, eucalyptus, rosemary, lavender and star anise, have been investigated in view of their use as repellent agents; the pomegranate peel extract has been used as stabilizing and reducing agent for the nanoparticles synthesis. The extraction of all essentials oils and pomegranate peel was done using microwave & ultrasound assisted extraction. All obtained nanomaterials were characterized by dynamic light scattering (DLS), scanning microscopy (SEM) and FTIR methods.

The nanomaterials were used in functionalization of textile materials for clothes fabrications, as ecological method for control of mosquito vectors.

metal nanoparticles; mosquito vectors; simultaneous microwave & ultrasound-assisted process

[A] http://www.who.int/denguecontrol/control_strategies/en/

Controlled release systems for rosemary and lavender essential oils obtained through microwave-assisted extraction

POSTER 27.07.2016 14:15 e-Poster Session (Show Room)

Raul-Augustin Mitran[A], Irina Atkinson[A], Jeanina Pandele-Cusu[A], Adriana Rusu[A], Victor Fruth[A], Mariana Patrascu[B]

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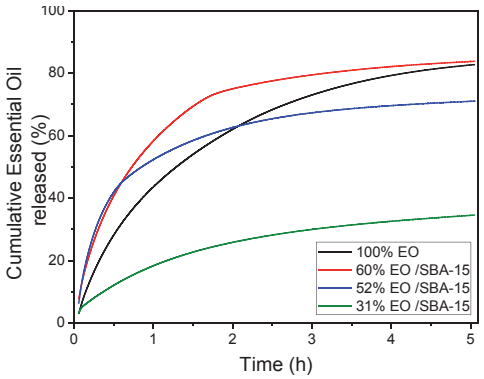
Essential oils (EO) can be obtained by extraction of volatiles compounds from plants. These oils contain secondary plant metabolites which often possess antimicrobial activity. As such, there has been a growing interest in employing these biorenewable resources as food preservatives. [A] One aspect related to optimizing the EO activity as food preservatives involves controlling the oils release rate into the medium. Second aspect presented in this paper is the microwave – assisted extraction of essential oils and their characterization method by GC/MS. Here we report a novel strategy to obtained controlled release systems for rosemary and lavender essential oils, based on encapsulation into high porosity matrices.

Controlled release systems were obtained by impregnation of rosemary and lavender Essential Oil into SBA-15 mesoporous silica (specific surface area 1000 m^2/g; average BJH pore diameter 6.82 nm; total pore volume 1.34 cm^3/g). The resulting materials were characterized by X-ray diffraction, FT-IR spectroscopy and thermogravimetric - differential thermal analysis. The influence of the EO loading on the release profiles was studied through isothermal thermogravimetric analyses in air, at 60°C. The essential oil release rate was found to depend strongly on its mass fraction in the resulting composite. Samples with low EO loading (<40% wt.) exhibit slower release rates than the pristine oil, while higher oil loading results in a two-step release profile, with faster initial release ("burst" release) followed by a sustained-release regime. This effect could be explained by the existence of EO molecules both nanoconfined inside the matrix mesopores and in the interparticle space. In particular, the EO-SBA-15 composite sample containing 31% wt. EO shows the highest potential as a controlled release system, with an EO release 2.5 times slower than pristine oil.



▲ Accelerated EO release profiles

[A] M. Hyldgaard, T. Mygind and R. L. Meyer, Frontiers in Microbiology, 3 (2012), 12.

essential oil; mesoporous silica; controlled release; microwave-assisted extraction

V-doped TiO_2 nanopowders obtained by microwave assisted sol-gel method

27.07.2016 14:15 e-Poster Session (Show Room)

Luminita Predoana [A], Irina Stanciu [AB], Jeanina Pandele [A], Silviu Preda [A], Adriana Rusu [A], Crina Anastasescu [A], Mariana Patrascu [C], Maria Zaharescu [A]

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Amog the methods used for preparation of TiO_2 and vanadium doped TiO_2 nanopowders, sol-gel technique is an efficient and versatile method. By combining the sol-gel method with microwave irradiation, the synthesis time and temperature is greatly reduced. In addition, microwave irradiation is clean, environmentally friendly, inexpensive and allows control of particle size, degree of crystallinity, and nanoparticles morphology [A][B].

In the present work, is discused the microwave influence on the catalytic properties of the vanadium doped TiO_2 nanopowders.

V-doped TiO_2 nanopowders with TiO_2:V_2O_5 molar ratios 98:2 and 99.95:0.05 were prepared by sol-gel and microwave (MW) assisted sol-gel methods. The reagents used were tetraethyl orthotitanate, vanadyl acetylacetonate and ethanol as solvent. The starting solutions were homogenized by stirring for 2 hours or were exposed to microwave (MW) for 5 min at 300 W into an oven operating at a frequency of 2.45 GHz with a maximum power of 2000 W. Reaction took place at the constant temperature of 80 °C for 5 min. Microwave (MW) power reached the maximum of about 300 W in the beginning of the reaction. After the ramp temperature of 80 °C was reached, the MW power decreased and stayed constant. The microwaves power used in the sol-gel synthesis was selected based on the literature data [C].

In order to eliminate the water and organic residues and to get crystallized nanopowders, the gels were thermally treated at 300℃ or at 450℃ for 1 hour, with a heating rate of 1℃ /min.

The nanopowders obtained were characterized from the point of view of their structure, morphology and adsorption properties and also were tested as photocatalyst for water splitting and oxidation of methanol.

[A] I. Stanciu, et al, Structure and properties of vanadium doped TiO2 powders prepared by sol-gel method, Rev. Roum. Chim., vol. 59, no. 11-12, pp. 919-929, 2014
[B] L. Predoana, et al, Structure and properties of the V-doped TiO2 thin films obtained by sol-gel and microwave assisted sol-gel methods, J. Sol-Gel Sci. Techn., doi: 10.1007/s10971-016-3972-9, 2016
[C] E. Vigil, et al, TiO2 thin films grown using microwave-activated solutions, J. Mat. Sci. Lette., vol. 18, pp. 1067-1069, 1999

V-doped TiO_2; MW assisted sol-gel method; catalytic properties; water splitting; methanol oxidation

Cannabis sativa ecosustainable microwave assisted extraction for cosmetic and nutraceutical applications

27.07.2016 14:15 e-Poster Session (Show Room)

C. Villa[A], C. Lacapra[A], R. Boggia[A], M. G. Signorello[A], M. Scovazzi[B]
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Hemp (Cannabis sativa (L.) is a C3 crop native to Central-Northeast Asia where there is evidence of its cultivation dating back over 5000 years ago. It is well adapted to the geography, soil and climate in Europe and it makes few demands on the soil and can generally be grown without the aid of chemical crop protection agents . As such it is an ideal candidate for integrated production (IP) and from an agronomic point of view, the prospects for the successful reintroduction of industrial Cannabis production are excellent. Therefore Cannabis can be considered a promising plant which is easy to grow and provides a high quality raw material [A].

Despite the great interest on Cannabis for its main cannabinoid tetrahydrocannabinol (THC), the fiber grade of this plant (with a very low content of THC, less than 0.3%) is cultivated for industrial purposes: flowers, seeds, fatty and volatile oils, and other parts of the hemp plant are currently used to manufacture foodstuffs and cosmetics.

Taking into account all the statements and our interest in green bioactive extracts from renewable sources [B][C], the aim of this work was the development of eco-sustainable microwave assisted methods to obtain industrial hemp extracts of cosmetic, nutraceutical and pharmaceutical interest.

Different solventless MAE (microwave assisted extraction) techniques, such as solvent-free microwave extraction (SFME) and microwave hydro-diffusion and gravity (MHG) [D][E], were exploited and the results compared to conventional methods in terms of efficiency and quality. The microwave applicator used for this study consists of a multipurpose prototype (multimode cavity) equipped with a specially designed Pyrex reactor, two optical fibers for temperature measurement and a control unit which allows managing and varying different parameters of the process such as emitted power, time and temperature. The furnace

presents two holes on the walls of the cavity which allow exploiting the prototype applicator for several applications, also for continuous flow reactions/extractions.

The green extracts obtained were evaluated for their biological potential and safety by means of in vitro assays. Methods and results will be reported.

microwave assisted extraction; cosmetics; green extracts; solvent-free; nutraceutical

[A] Cannabis Report of the Swiss Federal Commission For Drug Issues (EKDF)
[B] Villa C. et al. International Journal of Cosmetic Science 31, 55-61 (2009).
[C] Villa C., Boggia R., et al. EXTECH 2012, September 24-26, Messina, Italy (2012).
[D] Chemat F. and Cravotto G. Microwave-assisted Extraction for Bioactive Compounds, Theory and Practice. Eds., Springer, Food Engineering Series, New York, 2013.
[E] Chemat F., Vian M.A. and Cravotto G. Int. J. Mol. Sci. 2012,13, 8615-8627.

Topic 09: Microwaves in everyday life

Avoiding injury from microwavable products: an expert witness' suggestions

PLENARY 28.07.2016 09:40 Main Room

R. F. Schiffmann
R.F. Schiffmann Associates, Inc., New York, USA

Tools utilized for microwave cooking instruction development and validation

PLENARY 28.07.2016 10:00 Main Room

Dhawan Sumeet
Nestlé R&D, United States

Comparative study and simulation of *ex-vivo* tumor cell inactivation by microwave and conventional heating

ORAL 28.07.2016 11:15 Session B (Room 305)

Andreas Rosin[A], Corinna Drescher[A], Magdalena Suntinger[A], Thorsten Gerdes[A], Monika Willert-Porada[A], Benjamin Frey[B], Udo Gaipl[B]
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[B] University of Nürnberg-Erlangen, University hospital, Department of Radiation Oncology, Germany

A new trend in cancer therapy is the generation of an auto-immune response against tumors by cell-based vaccines. The combination of conventional and immune therapy could yield a systemic approach controlling residual disease and hidden metastases, achieving a long-lasting anti-tumor effect [A][B]. One example for anti-tumor vaccines are whole tumor cell-based vaccines (WTCV). WTCV offer a variety of tumor associated antigens which reduce the risk of tumor escape [A]. Autologous WTCV were one of the first anti-cancer vaccines tested. Patient-derived tumor cells were inactivated ex-vivo and re-injected to the same patient [C]. There are several studies investigating the effectiveness of WTCV for prostate cancer or melanoma [D]. The major requirement to WTCV is the complete inactivation of tumor cells and a significant increase of the immunogenic potential.

The present study investigates the potential of thermal inactivation of melanoma cells as WTCV and compares the results of microwave and conventional heating. For this purpose, a lab-scale closed loop reactor was designed that allows controlling the heating conditions in terms of inlet and outlet temperatures, flow rates and heating times. Experimental tests basically show that the rate of inactivation is dependent on temperature, flow rate and treatment time. But they also show that the microwave treated cells reach higher rates of inactivation compared to conventional heating at the same treatment conditions. In order to evaluate the effects of thermal treatment in detail, the temperature profiles for both, microwave and conventional, heating means were calculated and validated by numerical

simulation applying the same boundary conditions applied in the experiments. Simulations were carried out with COMSOL Multiphysics©. Axial and radial profiles for microwave and conventional heating showed quite comparable trends. In order to evaluate the thermal effects of the treatment, kinetics for protein denaturation were added, the space-time yield for denaturation was calculated and compared to experimental results. As matter of fact thermally triggered protein denaturation cannot exclusively explain increased cell inactivation during microwave treatment. Scanning electron microscope images of microwave-treated cells show highly perforated membranes in comparison to conventionally treated cells at the same conditions. The paper will discuss different reasons for the observed effect of cell inactivation by microwaves.

[A] C. A. Janeway, K. Murphy, K., P. Travers & M. Walport, Janeway's immunobiology. Garland Science Taylor and Francis Group, New York & London (2008).
[B] E.-M. Weiss, R. Wunderlich, N. Ebel, Y. Rubner, E. Schlücker, R. Meyer-Pittroff, O. J. Ott, R. Fietkau, U. S. Gaipl and B. Frey, "Selected anti-tumor vaccines merit a place in multimodal tumor therapies," Frontiers in oncology, vol. 2, no. 132, p. 1–11 (2012).
[C] C. Guo, M. H. Manjili, J. R. Subjeck, D. Sarkar, P. B. Fisher, and X.-Y. Wang, "Therapeutic cancer vaccines: past, present, and future," Advances in cancer research, vol. 119, p. 421–475 (2013).
[D] T. D. de Gruijl, A. J. M. van den Eertwegh, H. M. Pinedo, and R. J. Scheper, "Whole-cell cancer vaccination: from autologous to allogeneic tumor- and dendritic cell-based vaccines," Cancer immunology, immunotherapy, vol. 57, no 10, p. 1569–1577 (2008).

Measurements and analysis of electromagnetic shielding properties of a conductive polymer double shield

ORAL 28.07.2016 11:30 Session B (Room 305)

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[C] Dept. de Tecnologías de la Información y las Comunicaciones, Universidad Politécnica de Cartagena.

Electromagnetic shielding is the process of limiting the flow of electromagnetic fields between two locations by a barrier. An electromagnetic shield made of conductive polymer and conducting materials can be designed to achieve a specific required shielding performance. In this paper we measure the reflection loss, the absorption loss and the shielding effectiveness of, first; a single sheet of conductive polymer and second; a dual layer constructed of conductive polymer associated to aluminum. The material parameters conductivity and the thickness of the materials including conducting polymers will be introduced. The reflection loss, the absorption loss and the shielding effectiveness of single and double shield will be calculated using the transmission line matrix formalism. Experimental validation of mathematical simulations is carried out. Measurements have been carried out over a 10 MHz to 26 GHz frequency band. The measurements of the

electromagnetic shielding properties of single and double shield shows that the shielding effectiveness of a conductive polymer can be improved when it associated with a thin layer of aluminum and becomes higher than 40 dB, quantity required for many commercial shielding applications.

Problems heating small samples in domestic microwave ovens

28.07.2016 11:45 Session B (Room 305)

Robert F. Schiffmann
R.F. Schiffmann Associates, Inc., New York, USA

While investigating the cause of the serious burn injury to a consumer when using a product designated for microwave use, a series of test was run in various microwave ovens, including the specific oven involved in this case. During these tests it was determined that the location of the sample on a glass turntable has a strong influence upon efficacy of microwave coupling that may produce serious “hot spots”. i.e. intense heating at one location and very little heating at another. We noted that in one microwave oven a sample heated for one-minute near the edge of its of the glass turntable heated to only 63℃, but to over 230℃ in the center, above the drive shaft. In another oven the situation was reversed, with the ”hot” area being near the outer edge of the turntable. This effect is especially serious in small samples and may cause burn injuries. This paper will examine the positional effects in several domestic microwave ovens, both with and without turntables. These results imply that serious consideration must be made regarding the position within a microwave oven when testing any product or material, particularly if it is of relatively small size.

microwave ovens; turntables; small samples; microwave coupling; sample placement.

[A] R.F. Schiffmann, Problems in standardizing microwave oven performance, Microwave World, vol. 11, no. 3, pp. 20-24, 1980.
[B] G. Hooper, Food reheating instructions – a key element in delivery of safe, high quality foods, Campden BRI Report, 2014.

Topic 10: Modeling and numerical techniques

Microwave drying kinetics and infusion characteristics of olive leaves

ORAL 26.07.2016 16:45 Session A (Room 302)

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Leaves from olive trees are important byproducts of olive processing and oil industry which contain numerous phenolic compounds having strong radical scavenging activities. Leaves are rich in antioxidant compounds and has been used as a folk medicine against neuratic disorders and leaves extracts have anticancer, antihypotensive, antihypoglycemic, antimicrobial and antioxidant effects [A]. It is reported that oral administration of leave extracts reduce infarct volume, brain edema, blood-brain barrier permeability and improves neurologic deficit scores after transient middle cerebral artery occlusion in animals especially rats [B]. The pharmacological effects of leave extracts are attributed to the presence of polyphenols whose compounds in leaves are oleuropein, verbascoside, hydroxytyrosol, tyrosol, luteolin-7-glucoside and apigenin [C][D]. Flavonoids, rutin, catechin and luteolin have almost two and a half times more antioxidant activity than vitamin C, E and leave extracts show more antioxidant activity due to the synergy among flavonoids, oleuropeosides and phenols [E]. Olive leave and its extracts have been marketed as anti-aging, immunostimulator, anti-inflammatory, antioxidant, cardio protective, blood sugar regulating and antibiotic agents [F]. Olive leaves have been catching consumers' and researchers' great interest due to the these benefits on human health and the importance of phenolic compounds have made for researching this subject.

In this study, the olive leaves were dried by microwave method at different heating powers (90, 180 and 360 W) and drying kinetics of olive leaves examined to find the best mathematical model. Page model was the most suitable model rather than the others. Diffusion coefficients were ranged between $2.65 \cdot 10^{-10}$ to $6.87 \cdot 10^{-10}$ m^2/s and an increment in power level promoted the moisture diffusivities. Dried olive leaves were used to get leave tea and different infusion temperatures were examined to recover the total polyphenols (mg GA/kg) and radical scavenging activities (%). Rising in infusion temperature, especially at 100℃ enhanced the extraction levels of polyphenols from leave tea. Olive leaves dried at 360 W had higher phenolic contents (2282.9 mg GA/kg) among all samples. Microwave heating power did not have an important effect on radical scavenging activities of olive leave teas except 90 W.

microwave drying; mathematical modeling; olive leave; tea; infusion

[A] Mkaouar S, Bahloul N, Gelicus A, Allaf K, Kechaou N (2015). Instant controlled pressure drop texturing for intensifying ethanol solvent extraction of olive leaf polyphenols. Sep Purif Technol 145:139-146.
[B] Mohaghegi F, Bigdeli MR, Rasoulian B, Hashemi P, Pour MR (2011). The Neuroprotective effect of olive leaf extract is related to improved blood-brain barrier permeability and brain edema in rat with experimental focal cerebral ischemia. Phytomedicine 18:170-175.
[C] Yorulmaz A, Erinc H, Tekin A (2013). Changes in olive and olive oil characteristics during maturation. J Am Oil Chem Soc 90:647-658.
[D] Arslan D (2012).Physico-chemical characteristics of olive fruits of Turkish varieties from the province of Hatay. Grasas Y Aceites, 63(2):158-166.
[E] Sahin S, Ilbay Z, Kırbaslar SI (2015). Study on optimum extraction conditions for olive leaf extracts rich in polyphenols and flavonoid. Sep Sci Technol 50:1181-1189.
[F] Visioli F, Galli C (1998). Olive oil phenols and their potential effects on human health. J Agric Food Chem 46 (10):4292-4296.

Finite element modeling of microwave tempering and experimental validation

26.07.2016 17:00 Session A (Room 302)

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Dept. of Food Engineering, Mersin University, Mersin, Turkey.

The objective of this study was to develop and experimentally validate a finite element model for microwave tempering of frozen foods. A frozen block of shrimp was used as the model food. Microwave tempering in the cylindrical cavity of the system (operating frequency 915 MHz) was modeled using COMSOL Multiphysics and the RF module (Version 4.4). Temperature-dependent material properties (dielectric constant, dielectric loss factor, density, thermal conductivity and specific heat) were used in the model. Microwave tempering of the block of frozen shrimp (~2 kg) on a turntable was simulated at two different power levels (500 and 1000 W). Rotational movement of the block was included in the model by using a unique approach. Root mean square error (RMSE) values were calculated to compare the model results to the experimental temperature measurements. Temperature was measured using fiber optic probes at three different locations (center, edge, and corner). Surface temperature distributions predicted by the model and measured by a thermal camera were also compared. Close agreement was found between the model and the experimental results. The finite element model proved to be useful in optimizing the system and process design.

finite element model; microwave tempering; experimental validation

A simulation-based methodology for designing microwave heating processes

26.07.2016 18:00 Session A (Room 302)

Andreas Rosin, Monika Willert-Porada
University of Bayreuth, Chair of Materials Processing, Bayreuth, Germany

The objective of this work was to define material-specific engineering parameters, which allow to categorize the efficiency of high-temperature microwave processes for a wide range of temperatures, and to develop a guideline which supports the process engineer in designing a microwave heating process. For this purpose, the present study introduces a two-stage engineering procedure based on analytical and numerical calculations [A].

The assumed advantage of microwave heating of a volumetric heating effect for dielectric materials in comparison to conventional heating methods can be used in principle for fast dissipation of heat in materials with poor or moderate thermal conductivity. First of all, the absorbed microwave power is dependent on permittivity, which is a function of material composition, density, internal structure and temperature level. Secondly, power absorption is based on the object's shape, resonator geometry and process environment.

In order to categorize the material-specific microwave absorption dependent on permittivity and object size, a method is presented based on simplified characteristic numbers, which allow a process engineer an early evaluation of energetic efficiency regarding the conversion of microwave energy into heat over a wide temperature range based on permittivity. In order to review the material-specific aspects of microwave heating three model substances were selected. Each temperature-dependent permittivity shows a characteristic behavior: (a) Al_2O_3 shows a moderate increase of dielectric loss, (b) ZrO_2 shows strong increase of dielectric loss with temperature, and (c) silicon carbide shows low temperature-dependent increase of dielectric loss at high permittivity level. With help of these materials a macroscopic multi-scale investigation of microwave absorption was performed for spherical objects ranging from Millimeter to Meter scale. As a fast analytic method the calculation of energy absorption was based on Mie theory. Based on these calculations simplified characteristic numbers for evaluation of high-temperature microwave processes could be compiled for rendering nomograms.

The second stage of microwave process engineering could include numerical procedures like FDTD and FEM. Sintering of ceramic materials was selected as example. The objective of the presented simulations is evaluation and optimization of energy balance of the processes by combination of different heat sources like microwave heating at 2.45 GHz, resistive heating, and passive infrared radiators (susceptors). For this aim a procedure for coupling of microwave and thermal simulation was developed and tested that allows considering changes in sample size typical for sintering of ceramic materials, and is aiming at realizing adequate energy efficiency and process stability over the complete temperature range of the sintering process. Furthermore, process characterization was extended by

calculation of thermal shock parameters in order to evaluate the mechanical integrity at high heating rates.

[A] A. Rosin, Dissertation, University of Bayreuth, Department Engineering Science, "Entwicklung einer simulationsgestützten Methodik zur Auslegung von Hochtemperatur-Mikrowellenprozessen" (in German), 2015, https://epub.uni-bayreuth.de/1914/

CAD of a dielectric insert supporting uniformity of microwave heating

26.07.2016 18:15 Session A (Room 302)

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While microwave heating is known for its ability to improve the efficiency and quality of a variety of applied thermal processes, the intrinsic non-uniformity of microwave-induced temperature fields remains the key issue challenging the design of many practical applicators. Numerous attempts aiming to homogenize the heat release have been reported. It has also been shown [A] how with the use of the method of optimal material design to determine the position and micro-geometry of composite dielectric layers that ensure uniformity of the electric field of the TE_{10} mode within the rectangular dielectric block in a rectangular waveguide.

In this paper, going well beyond the concept of the approach [A], we propose a technique for determination of parameters (geometry and complex permittivity) of a homogeneous dielectric insert which, when placed inside a microwave applicator, homogenizes distribution of dissipated power in the processed material. The technique is based on full-wave computational analysis of the applicator and identification of geometrical parameters of the insert that, along with its dielectric constant and the loss factor, are suitable for being design variables of the optimization problem. The latter is formulated for an objective function linked with uniformity (and the relative standard deviation (RSD) of dissipated power serving as a uniformity metric) and solved with the sequential quadratic programming. The CAD procedure outputs the shape and complex permittivity of the insert that minimize the RSD and thus make the heating pattern as uniform as possible.

Functionality of the developed technique is illustrated by synthesis of a rectangular dielectric block in a 2.45 GHz applicator for microwave fixation [B]. The insert is expected to ensure uniformity of heat release in its internal conical space filled with the medium of complex permittivity $\varepsilon = 46.9 - j7.2$ typical for a mouse brain (grey tissue). Four optimization runs (started from different initial conditions) result in the patterns characterized by the RSD equal to 19.1, 19.3, 20.5 and 27.5%; the best (19.1%) one is produced by the block with $\varepsilon = 52.67 - j2.53$. For comparison: four non-optimized (randomly chosen) sets of design variables ensure the patterns with the RSD between 60 and 135%.

While the illustrative example evidently demonstrates that the "quality" of homogenization of dissipated power strongly depends on the chosen optimization space, it is clear that the proposed technique may be practical in designing a variety of applicators with high rates of microwave thermal processing.

complex permittivity; dissipated power; homogenization; modelling; optimization; relative standard deviation; sequential quadratic programming

A K.A. Lurie and V.V. Yakovlev, Control over electric field in traveling wave applicators, J. Engng Math, vol. 44, no. 2, pp. 107-123, 2002.

B E.M. Moon, J.F. Gerling, C.W. Scouten, and V.V. Yakovlev, Modeling-based optimization of thermal processing in microwave fixation, Proc. 49th IMPI's Microwave Power Symp. (San Diego, CA, June 2015), pp. 54-55.

Analysis and design of multimode cavities including mode stirrers

26.07.2016 18:30 Session A (Room 302)

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In this work, the cavity-shape perturbation theory has been used in order to study the effect of the introduction of a mode stirrer into a multimode microwave cavity. Based on this perturbation theory, a new method has been developed in order to design a multimode microwave cavity that contains a mode stirrer.

This semi-analytical design method is based on the application of the perturbation theory and a genetic-algorithm optimization procedure. The variable parameters of the genetic-algorithm are the dimensions of the cavity (a, b, L) and the goal is to allocate some specific resonant modes within the 2.44-2.46 GHz frequency range.

The cavity dimensions were set around 40x20x40 cm^3. Analytically, some modes could be excited within the working frequency range of the source (2.4-2.5 GHz) for the empty cavity. Specifically, four degenerated modes are supposed to be allocated at 2.4286 GHz (TE_{125}, TM_{125}, TE_{521}, and TM_{521}), thus those modes were selected for their study.

Both the cavity-shape perturbation theory and the proposed design method have been validated with CST Microwave Studio commercial software. The obtained results reveal that the proposed procedure may be used with a mean relative error of 0.1027 % in the resonant frequency estimation, thus facilitating the multimode cavity design.

perturbation theory; stirrer; microwave heating; multimode microwave applicators

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Multiphysics numerical modeling of microwave heated porous catalyst bed for biofuel production using COMSOL

ORAL 26.07.2016 18:45 Session A (Room 302)

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Due to the potential advantages, microwave technology is now extensively studied to investigate the effect of microwave heating of catalyst in endothermic reactions. In order to design an experimental set up for microwave heating of catalyst, it is important to understand how the catalyst can be heated in the reactor. The dielectric properties of HZSM-5 powder were measured at nine temperatures ranging from 250-700 K. A preliminary numerical model was developed using COMSOL Multiphysics 5.1 studying the effect of various catalyst bed shapes, sizes and positions within the reactor on the microwave heating of porous catalyst bed. Two shapes of catalyst bed a) block and, b) cylindrical and five different positions a) center of the waveguide, b) base of the waveguide (z = –0.03 m), c) upper edge of the waveguide z = 0.03 m, d) z = –0.01 m and e) z = –0.02 m were studied. Secondary model was developed where the flow of gas through the reactor was taken into account. The secondary model compared the heat and flow profiles for microwave and conventional heating of catalyst bed. The data was also validated with the experimental results. It was observed that the sample position, shape and size of the sample, all significantly affect the heating profile and temperature gradient inside the porous media heated using microwave irradiation. The experimental results and the predicted temperatures were a close match. Microwave heating had higher total internal energy but conventional heating had lower temperature gradient after reaching steady state. This type of numerical modeling can be used to optimize the process parameters and design an efficient process depending on the ease of operation, manufacturing, and cost efficiency.

Numerical and experimental investigation of continuous-flow microwave heating in a cylindrical applicator

POSTER 26.07.2016 14:15 e-Poster Session (Show Room)

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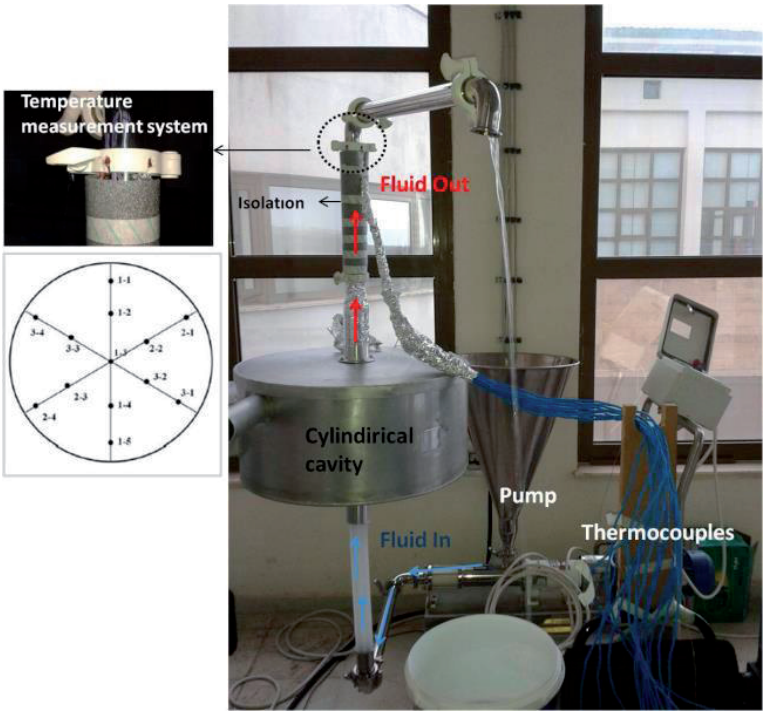
In this study, heating of fluid foods in a specially designed continuous-flow microwave heating unit (915 MHz and 5 kW maximum power) was simulated by finite element method using COMSOL Multiphysics. Experiments were then conducted with different test fluids (distilled water, 0.5% and 1% CMC solutions) at a power of 4 kW and different flow rates (1, 2, 3 L/min) to validate the model. The pilot-scale unit consisted of a straight PTFE tubing (diameter 38 mm, microwave exposure length 254 mm), within the cylindrical cavity of the unit (see figure). Cross-sectional temperature distribution at the exit of cavity was obtained by measuring temperature at thirteen different points by thermocouples distributed across the cross-section of tubing.

Low temperature increases achieved during the experiments showed that microwave energy was used inefficiently in the current setup. The highest temperature increase (12℃) was observed in case of 1% CMC



◀Continuous-flow microwave system and thermocouple assembly showing thermocouple locations

Topic 11: Nanotechnology

A novel microwave assisted precipitation technique for nanonization of nutraceuticals

29.07.2016 09:20 Main Room

Atidya Nayak
University of Birmingham

Due to lack of a sustainable and scalable approach for manufacturing the shape-engineered nanocrystals of organic molecules (bioactives e.g. polyphenols; and pharmaceuticals), their use in the food and pharma sectors is trivial. Hence there is a need for an innovative and novel technology to fill this gap.

In this regard, recently we have invented a novel nanonization technology, which is based on the use of microwave energy. In principle, microwave energy is applied to rapidly and uniformly evaporate solvent from the solvent and antisolvent mixture using their dielectric properties to precipitate the solute to induce nucleation & crystal growth. Due to higher automation of microwave instruments, nucleation ambience e.g. temperature, vacuum, pressure etc., that affects the particle shape are controlled by controlling the reaction composition, processing parameters and operating parameters.

This innovative technology is expected to bring a step change in the state-of-the-art crystal engineering.

In-situ emission spectrophotometric analysis of TiO_{2-x} during microwave irradiation

27.07.2016 18:45 Session B (Room 305)

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Microwave non-thermal effects have been reported in these days, however, the effects are still discussing. The authors was reported that the reduction reaction of copper oxide was enhanced by microwave H-field irradiation compared to conventional heating [A]. The activation energy in H-field irradiation was decreased to 1/3 compared to that in conventional heating. As the sample was reduced to Cu_2O, microwave energy is transformed directly into reduction energy because the enthalpy of the reduction reaction is larger than activation energies in H-field heating. The reduction enhancement was observed in the case of TiO_{2-x} [B]. Although the enhancement effect was observed, the mechanism did not elucidated. If microwave energy can be transformed directly into the reduction energy, it can also be transformed directly into light emission. In TiO_{2-x} case, the activation energy which was directly supplied from microwave energy was about 300

kJ/mol, i.e. 3 eV. Thus we tried to detect the directly supplied energy by *in-situ* emission spectrophotometric analysis during microwave irradiation and explain the mechanism of microwave non-thermal reduction-enhancement effect. Partially reduced TiO_2 (TiO_{2-x}) was irradiated with microwave at the maximum point of H-field by using single-mode cavity under high vacuum state (about 10^{-4} Pa). During irradiation, *in-situ* emission spectrophotometric analysis was conducted. From the experimental results, the authors detected some line spectra assigned to Ti I (498.173 nm and 521.039 nm), which was distinguished from continuous spectrum (thermal radiation). The result suggests that the microwave energy was integrated in the materials and the integrated energy excited TiO_{2-x} before thermalization.

non-thermal microwave effect; emission spectrum; activation energy

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Microwave-assisted synthesis of Cu_2ZnSnS_4 (CZTS) nanocrystals for photovoltaic application

28.07.2016 14:15 e-Poster Session (Show Room)

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Nowadays, the world demand for electrical energy is huge and it is still increasing. One of the alternative ways to provide this problem are photovoltaic devices (PV) like solar cells, but their cost of production is still too high compared to their efficiency [A]. In order to reduce this cost intensive scientists research are conducted on the new generation of functional materials as cheaper replacement for silicon-based solar panels. One of the most promising materials for PV application is Cu_2ZnSnS_4 (CZTS) due to relatively narrow band gap (about 1,5 eV) and high absorption coefficient (about $10^4 cm^{-1}$) [B]. Typical PV devices based on CZTS consist of one continuous layer of CZTS crystals, but using the discontinous layer of nanocrystals could improve the efficiency of such devices [C]. There are a lots of methods to obtained CZTS films but there is no method with using microwave radiation [D]. The aim of this study was to obtained nanocrystals CZTS using a microwave reactor, which can be used for nanoparticles/conjugated polymer solar cells devices. The reactions were carried out in aqueous solution and organic solvents, respectively. In the first method as solvents the ethylenediamine and water were used. In the second method, the oleylamine or octylamine were used. The synthesis were carried in various temperature, under various pressure and with different time of reactions. The obtained materials were characterized using XRD, UV-Vis, FTIR and TEM methods. The XRD patterns and TEM images confirm crystal structures of CZTS nanoparticles. UV-Vis measurement shows that, the all materials

have absorbance in the spectrum of 900-300 nm. In conclusion, the microwave synthesis it could be promising alternative method to fabrication CZTS materials for optoelectronic application.

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Cu_2ZnSnS_4 (CZTS); synthesis under microwave irradiation; nanocrystals

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Topic 12: Process control

In-situ shrinkage-temperature measurement during microwave sintering: a useful tool to control the overall densification process

ORAL 27.07.2016 18:00 Session B (Room 305)

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Microwave energy is a very efficient way for the synthesis of new materials or the sintering of ceramics. In the last years, many improvements have been realized, including the design of original susceptors and the discovery of the most suitable thermal insulator among others. All these developments were carried out to optimize the conversion of electromagnetic energy into heat. However, most of the performed developments result by trial and error methods until now. This strategy can be time consuming and is not the most suitable way to optimize any processes. With the intention to produce high quality sintered oxides parts using microwave energy as a source of heating, we have designed specific assemblies including thermal insulating materials and eventually SiC or ZrO_2 based susceptors. These assemblies were designed based on data obtained by modelling the electrical field distribution, as a function of the assembly. In addition, it has been developed a specific instrumentation to measure the shrinkage and the sample temperature during microwave sintering, in working carefully on the temperature calibration, using original calibration material. In this work, it will be presented the development of this specific instrumentation.as well as some applications. The as-developed instrumentation allows to record shrinkage – temperature curves during microwave sintering and these data can be exploited using various phenomenological sintering models such as Master Sintering Curve concept. This enables to compare in details the microwave sintering over the conventional process. It is also a useful method to investigate the effect of dopants on the microwave sintering of oxides, accordingly the microwave absorption properties of the dopant. Some examples will be presented showing successful sintering experiments, on different materials, based on the dilatometric measurements (nano-powders $MgAl_2O_4$, Al_2O_3), with different sizes and geometries. The microstructure (grain size-homogeneity) is also investigated as a function of the thermal history of the microwave heated samples, and the advantages of the microwave process over conventional are discussed.

microwave sintering; oxides; temperature calibration; shrinkage

Application of the *in-situ* shrinkage-temperature measurement during microwave sintering of doped spinel

ORAL 27.07.2016 18:15 Session B (Room 305)

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Transparent magnesium aluminate spinel ceramic presents very interesting optical properties, especially related to its excellent optical transmission from 0.2 to 5 μm, and attractive mechanical properties. Widely used in the refractory industry, this oxide is a promising candidate for optical and mechanical applications for structural applications (ballistic materials for instance). However, obtaining transparent materials requires a total elimination of porosity, and so implies a very good control of the sintering process. In this work, we have investigated the microwave sintering process in a view of getting homogenous $MgAl_2O_4$ microstructure, suitable for producing, after HIP, transparent ceramic material. For doing that, it has been developed a specific instrumentation to measure the shrinkage and the sample temperature during microwave sintering, in single mode 2.45 GHz microwave cavity, and in working carefully on the temperature calibration. In this work, it will be presented the shrinkage curves of the spinel material, being microwave sintered with different dopants (MgO, TiO_2 and ZrO_2). Those dopants were selected to promote densification through solid state sintering, in a view of getting optimal microstructure (low and fine inter-granular porosity/small grain size). In doing so, the specific effects of the dopants are investigated. The dilatometric data are also exploited through different phenomenological models (Master Sintering Curves and Constant Heating rate methods) in order to characterize as far as we can the sintering behavior of the spinel doped materials under microwave, in relation with their microstructures.

microwave sintering; spinel; shrinkage; master sintering curve

Scale-up of internally transmitted microwave assisted crystallizer for process control

ORAL 27.07.2016 18:30 Session B (Room 305)

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Controlling the size distribution of crystals during the crystallization process is one of the important control objectives. Direct nucleation control (DNC) is a feedback control strategy used for maintaining a narrow size distribution but removal of unwanted fine particles.

DNC is based on in situ measurement of number of particles and by utilizing temperature cycling a control over the number and size distribution of particles can be obtained by alternating between dissolution and growth cycles [A].

One of the major draw backs of the DNC is that the temperature cycles needed to dissolve the nuclei give rise to a considerable increase in the process/batch time in case a conventionally heated crystallizer (via the jacket)is used However recently it has been demonstrated that application of microwave heating, the same control performance can be achieved with a 50% reduction in process/batch time [B]. The latter experiments where performed in a 1 litre jacket vessel from which the slurry was circulated via an external loop through a microwave cavity. However for industrial application a crystallizer integrated with microwave generator is preferred so that the microwaves can be directly introduced into the crystallizer.

In this communication we present DNC experiments using a microwave integrated crystallizer utilizing the internal transmission line technology made available by Sairem SAS. Some of the key challenges addressed in this study are: Evaluation and optimization of the microwave heating to ensure homogeneous temperature distribution in the system, Optimization of the reactor configuration for safe introduction of PAT tools essential for DNC feedback (e.g FBRM), by avoiding the exposure to high intensity MW field and evaluation of the performance of microwave assisted DNC to meet the performance observed in lab scale trials.

scale up; microwave; crystallization; nucleation; control

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Topic 13: Radiation safety

Parameters stabilization for time-domain shielding effectiveness of enclosures in presence of a plane wave

27.07.2016 17:00 Session A (Room 302)

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Electromagnetic shielding of metallic enclosures with an aperture are simulated and measured in the frequency and time domain in this paper. Recently several new figures of shielding effectiveness (SE) have been proposed [A][B][C][D]. Much work has been done regarding numerical values [E], however little related to measurements has been carried out even the magnetic part. In this work, we get the simulated results for these SE definitions when an incident plane wave, with a determined bandwidth, excites the enclosure. The plane wave can be treated as a reference interference to compare with other cases. Measurements [F] and simulations are in good agreement. This study evaluates three selected parameters for the SE evaluations (Electric, Magnetic and Energy density) for several bandwidths. In this work, stabilized values for each these three parameters are looked for, and a first approach is defined for each parameter in order to enlarge the scope of the values and get values with a wider meaning. Finally, an example is represented for a sweep of the size of the apertures with these new approaches.

aperture; enclosure; plane wave; shielding effectiveness

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Peral Submarine:

The very first submarine to be electrically powered with underwater torpedo-firing facilities. Isaac Peral y Caballero (Cartagena, 1 June 1851 - 22 May 1895, Berlin), was a Spanish engineer, naval officer and designer of the Peral Submarine. He was the “father of the modern submarine”.