4 Energy Efficiency, Materials and Resources (EMR): Energy-Efficient Processes – Materials Processing with Microwaves –

4.1 Characterization of dielectric materials

4.1.1 In-situ dielectric characterization and calorimetry of pressurized chemical reaction

Owing to the selective and volumetric nature of heating the application of microwave enables faster and more energy efficient processing in various fields of applications such as for example microwave chemistry. For the successful design of microwave assisted chemical reactors, beside the knowledge of dielectric properties, calorimetric information as well as information about the reaction kinetics are important. Calorimetric measurements always imply samples under test being in thermodynamic equilibrium with its environment. Since in case of microwave heating this never happens, beside an accurate measurement of the microwave power, absorbed in the system, the detailed knowledge about its thermodynamic behavior is mandatory.

Till know only qualitative calorimetric measurements, combined with dielectric characterization in microwave cavities, had been reported. Here, the sensitive microwave power measurements were extended with heat transfer simulations to enable a more quantitative estimation of the power absorbed in the material under test.

To account for the heat transfer losses from the sample heated with microwave the COMSOL Multiphysics software package was employed (see Fig. 4.1.1). For that problem, the heat source defined as the microwave energy dissipated in the dielectric material, was estimated by means of electromagnetic simulations using CST Microwave Studio.

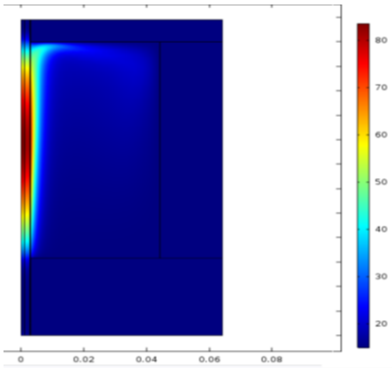

Fig. 4.1.1: COMSOL simulation of the temperature distribution in the cavity.

4.1.2 Ku-Band Fabry Perot-Resonator

To enable the dielectric characterization of low loss liquid as well as solid samples at frequencies close to the ISM Band at 24.125 GHz, a so called Fabry Perot Resonator has been designed and build (see Fig. 4.1.2).

This is an open resonator concept consisting of a planar and a spherical aluminum mirror. The cavity built reveals a measured quality factor of about 85000 that provides a resolution limit of the dielectric loss tangents at values as low as 10^{-4} as could be demonstrate for a low loss sample of CaF_2 single crystal (see Fig. 4.1.3). The planar mirror which holds the material under test can be heated to temperatures up to 150°C and therefore allows temperature depended in-situ measurements as well. The measurement procedure as well as the data interpretation is fully computer controlled, what allows a reproducible and comfortable data acquisition.

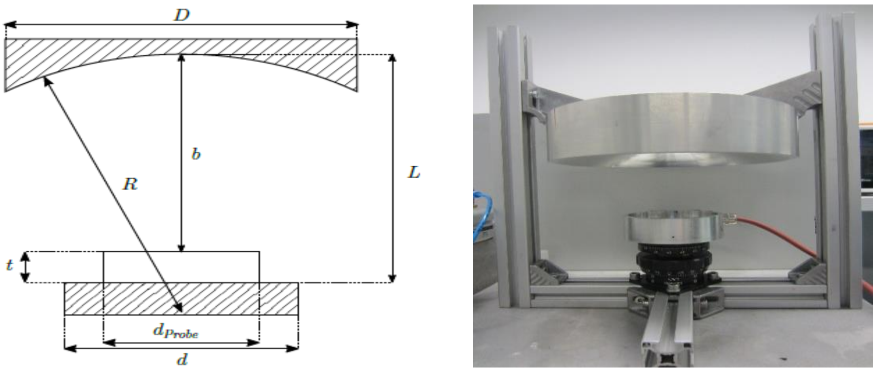

Fig. 4.1.2: Scheme (left) and photo (right) of the Ku-Band Fabry Perot resonator.

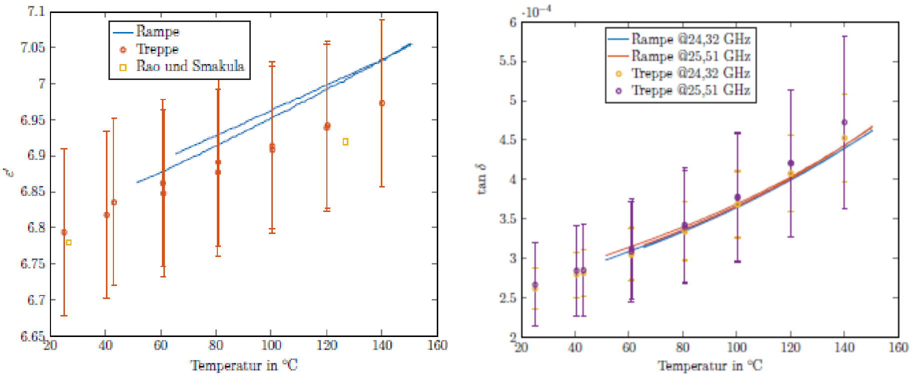

Fig. 4.1.3: Dielectric constant (left) and dielectric loss factor (right) of a CaF_2 single crystal as a function of temperature for continuous and stepwise heating at 24 GHz.

4.2 Plasma chemistry

End of 2016 a new lab was established for investigations on microwave assisted plasma chemistry. Most of the necessary infrastructure has already been installed (see Fig. 4.2.1). The first plasma and experimental investigations on plasma assisted CO_2 conversion are planned for 2017. For the perspective chemical reactions the optimal microwave sustained plasma scenarios and applicators will be developed. For that purpose the equipment of the new plasma laboratory is under development. Already now a 6 kW microwave plasma source which can operate at atmospheric pressure is available, that will allow plasma and gas temperatures up to 3500 K. For analysis of reaction products optical emission spectrometer Ocean Optics HR2000+ with the working wavelength rage of 200 to 1100 nm will be used. Mass flow controller will allow the precise control of feeding gases like argon, air, carbon dioxide and hydrogen. The laboratory is equipped with a system for removal of exhaust gases and a gas monitoring and alarm system.

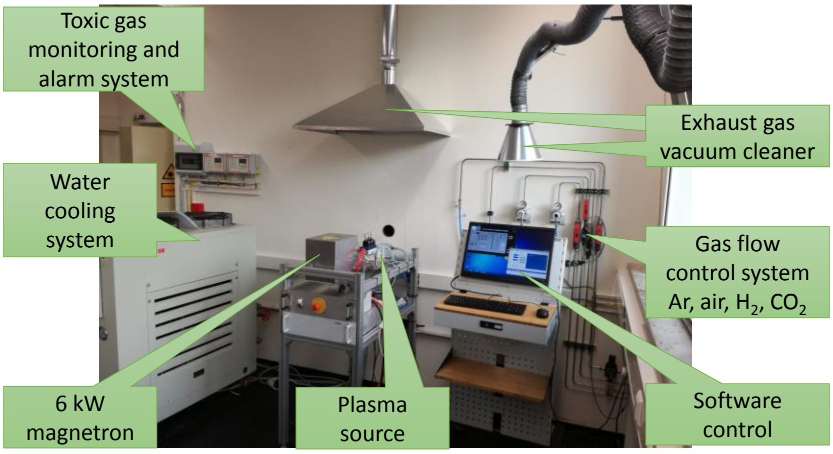

Fig. 4.2.1: Photo of the plasma laboratory.

4.3 Microwave assisted extraction of microalgae

The extraction of oil from micro algae provides an alternative, regenerative source of liquid fuels. First experiments on the treatment of algae cells with microwave radiation have motivated the development of an advanced microwave applicator operating in pulse regime at a frequency of 2.45 GHz. A short pulse of high power microwave being absorbed in the algae cells facilitates greatly the release of oil to the surrounding medium. For short microwave pulses heating of the surrounding medium is negligibly and the microwave energy is spent fast and for oil extraction solely. It makes such an approach very energy efficient and attractive for industrial application. The scheme of the experiment is presented in Fig. 4.3.1, where the main element is the microwave cavity (3). The magnetron source (1) isolated with a circulator (9) couples the wave into the cavity (3). The algae suspension (4) is pumped (2) through the cavity and collected (4) for further analysis. The temperature of the suspension (5) is controlled at the input and output of the cavity with thermocouples (5). The absorbed energy is measured by use of a directional coupler (8) and an oscilloscope (7).

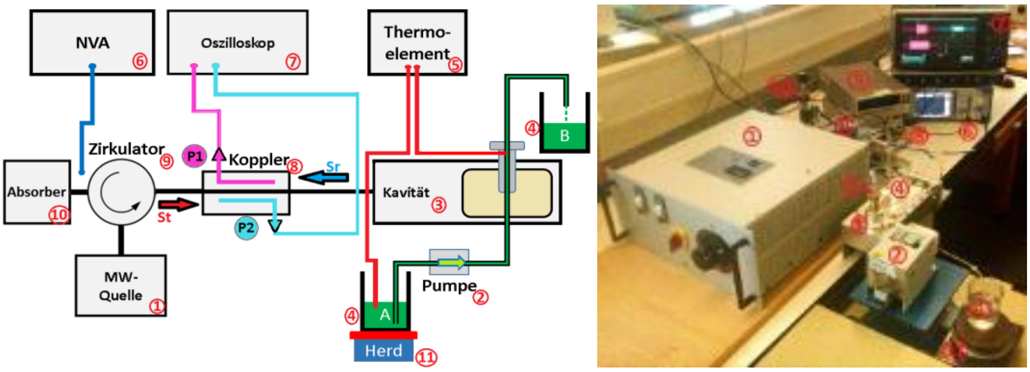

Fig. 4.3.1: Scheme of experiment on the oil extraction from algae by means of pulsed microwave radiation (left) and the photo of the experiment in the lab (right).

The microwave applicator design and fabrication was the central part of the present work. The design is based on an optimized cylindrical cavity operating at the TM_{010} mode. This provides the concentration of the microwave energy in the algae suspension flowing through the region where the electric field is maximal. Additionally the design features a tuning element allowing to shift the resonance frequency and to maximize the energy coupling into the algae dispersion for different dispersion media.

4.4 Collaboration Projects

4.4.3 Microwave assisted selective bonding

In the frame of a collaborative project with CARL MEISER GMBH & CO.KG on the microwave assisted bonding of thin decorative technical textiles to plastic substrates the model of the heat transfer and microwave absorption in the coating-glue-substrate sandwich structure (Fig. 4.4.1) was developed. This project has been funded by the Federal Ministry of Economy Affairs and Energy within the Central Innovation Programme for SMEs

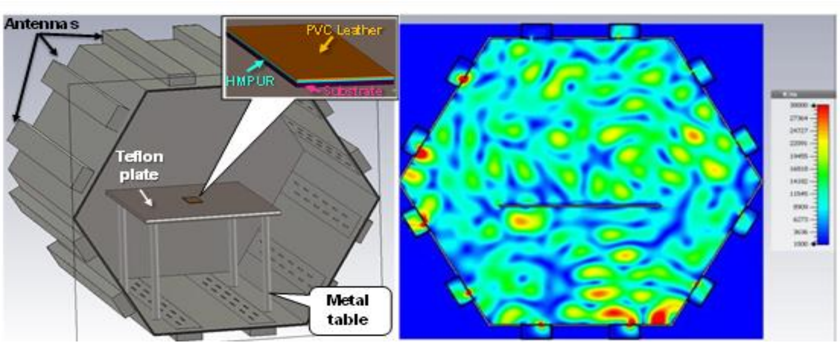

Fig. 4.4.1: Electromagnetic simulation of the HEPHAISITSOS VHM100/100 oven. Geometrie of the model (left), distribution of the electric field in a vertical cross section (right).

The project objective was the process optimization by variation of the microwave susceptibility of a polyurethane hot-melt (HMPUR) adhesive, the launched microwave power and the radiation time, respectively to enable a good selective heating within the sandwich structure (see Fig. 4.4.2) and a temperature profile, resulting in a high quality bonding.

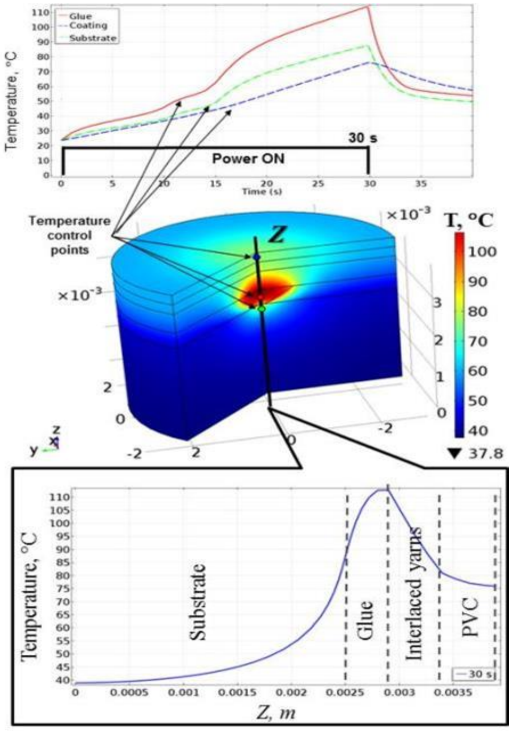

Fig. 4.4.2: Result on heat transfer modelling with COMSOL Multiphysics.

A variation of the HMPUR susceptibility has been achieved by blending it with carbon fibers in the range from 1 wt.% to 4 wt.%. The modelled coating was a PVC synthetic leather with a PET interlaced yarn textile layer at the wrong side. The substrate was a dense ABS-PC material. For process analysis the absorbed microwave power was estimated by use of CST Microwave Studio (Fig. 4.4.1). The resulting temperature fields inside the materials were modelled by use of COMSOL Multiphysics taking the results from CST simulations as heat source (Fig. 4.4.2).

After 30 seconds of heating the resulting temperature profile within the sandwich structure of only 4 mm in thickness enables the curing of the HMPUR adhesive at 110 °C whereas the coating material remains at lower temperatures with no thermal damaged. The temperature difference of 40° C between the glue and coating domains due to selective heating is remarkable, accounting the distance between them of about 1 mm only (see Fig. 4.4.2). For the found optimal heating scenario and glue formulation the microwave assisted coating of a Land Rover glove-box cover was successfully demonstrated (see photo in Fig. 4.4.3).

Fig. 4.4.3: Result on microwave assisted coating of Land Rover glove-box cover.

4.4.4 SYMBIOPTIMA

The KIT task within the H2020-SPIRE-2015 European project SYMBIOPTIMA is to support the development of an industrial scale microwave reactor for the recycling of PET plastic waste. The objective is the energy efficient microwave assisted chemical depolymerization of the polyethylene terephthalate (PET) into its monomers terephthalic acid (PTA) and ethylene glycol.

The basis for that design is a precise knowledge of the dielectric properties of the reactive mixture as well as the materials of the applicator within the working temperature range. For this purpose an advanced dielectric measurement setup for in-situ dielectric characterization of lossy liquids under microwave heating conditions and under pressure conditions had been developed. Here the cavity perturbation method based on the simultaneous excitation of two modes, TM_{010} and TE_{111}, at a frequency of about 2.45 GHz is applied. That allows the dielectric characterization of high permittivity materials with sufficient accuracy, not feasible with the classical analytical perturbation method. To do so the TM_{010}-mode was used to measure the dielectric constant and of the TE_{111}-mode for the dielectric loss, respectively. The following figure shows the designed test-set.

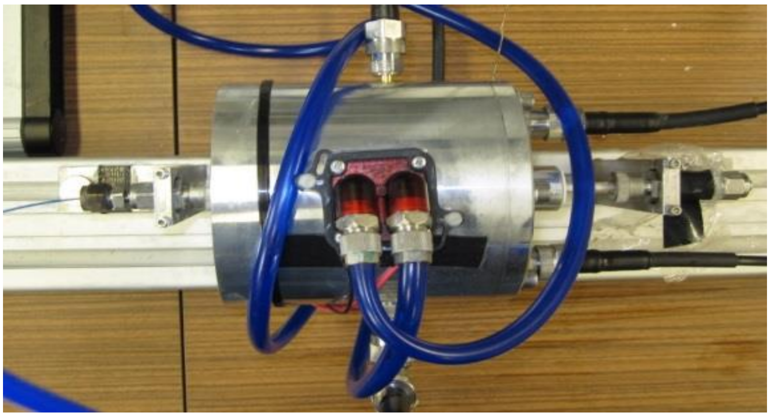

Fig. 4.4.4: Test setup for dielectric measurements.

The quartz tube sample holder was closed at both ends with Swagelok connectors, which can withstand a pressure of 18 bars. For active control and stabilization of the cavity temperature, two Peltier elements of type QC-241-1.0-3.9M were installed. The sample temperature was measured by use of a fibre optic sensor located in the centre of the sample.

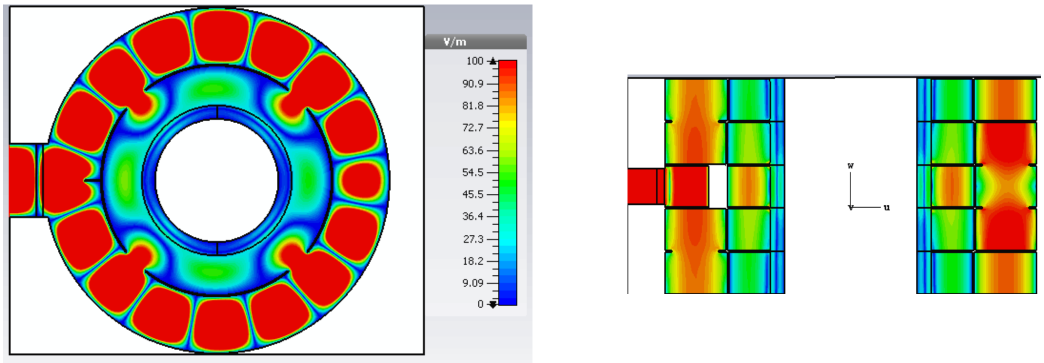

Fig. 4.4.5: Simulated field distribution in a tubular chemical reactor

Based on the measured dielectric properties and using common 3D software tools, an optimised industrial scale microwave applicator design has been developed and optimized. The achieved design offers a well-defined and even distribution of the power from a single magnetron source within the applicator and the process material, as can be shown from simulation results (see Fig. 4.4.5). This can be achieved by using a network based on rectangular waveguides, mutually connected by coupling ports that were optimized in size and position. This applicator design enables high field homogeneity in azimuthal as well as axial direction in a tubular reactor. At the same time the energy efficiency is high with less than 10 % reflected power in the overall range of expected material permittivities.

4.4.5 InnoConTemp

Microwave heating has a great potential to replace classical heating processes, such as convection or radiation heating. The main advantage is the possibility to heat in the whole volume of the product, therefore the slow thermal conduction doesn’t limit the heating process. Compared to a classical heating system both energy consumption and cycle time can be reduced. However, there aren’t a lot of microwave systems implemented in industry because of insufficient temperature uniformity in the product.

Therefore, the aim of the technology transfer project InnoConTeMP (Innovative Control of Temperature Distributions for Microwave heating Processes), which is co-financed by the KIT Innovation Fund and has started in September 2016, is to improve the temperature distribution of a workpiece in the HEPHAISTOS systems licensed to the industry partner Vötsch Industrietechnik GmbH. As a starting point some ideas of the FLAME project are used. The idea is to control the power levels of each distributed antenna in the HEPHAISTOS oven with an intelligent algorithm, so that the temperature distribution of the product is improved. One or more of the three different control algorithms, which were developed within the FLAME project, will be used and optimized: model predictive control (MPC), neural network control (NNC) and reinforcement learning control (RLC). The improvement compared to a classical PID controller, that only regulates the power levels of all microwave sources to the same value (no independent control of each source), is shown in Fig. 4.4.6. There's a much smaller temperature difference ΔT between all measured values by using the model predictive controller.

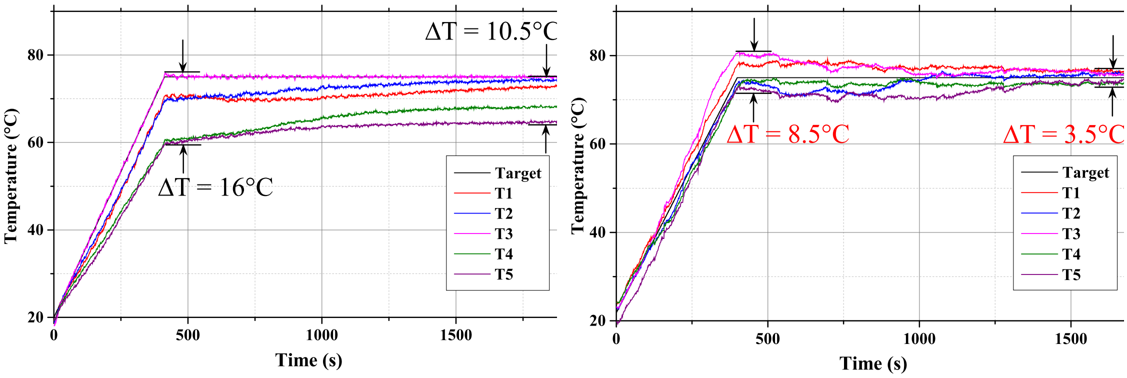

Fig. 4.4.6: Comparison of a PID (proportional–integral–derivative) controller (left) and an innovative model predictive controller (right).

At the end of the project the control algorithms will be implemented in the control software S!MPAC of our partner Vötsch, so that the HEPHAISTOS systems can be sold with an improved temperature homogeneity. This will allow to develop new applications where heating uniformity is more critical and could not be achieved with existing technology.

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