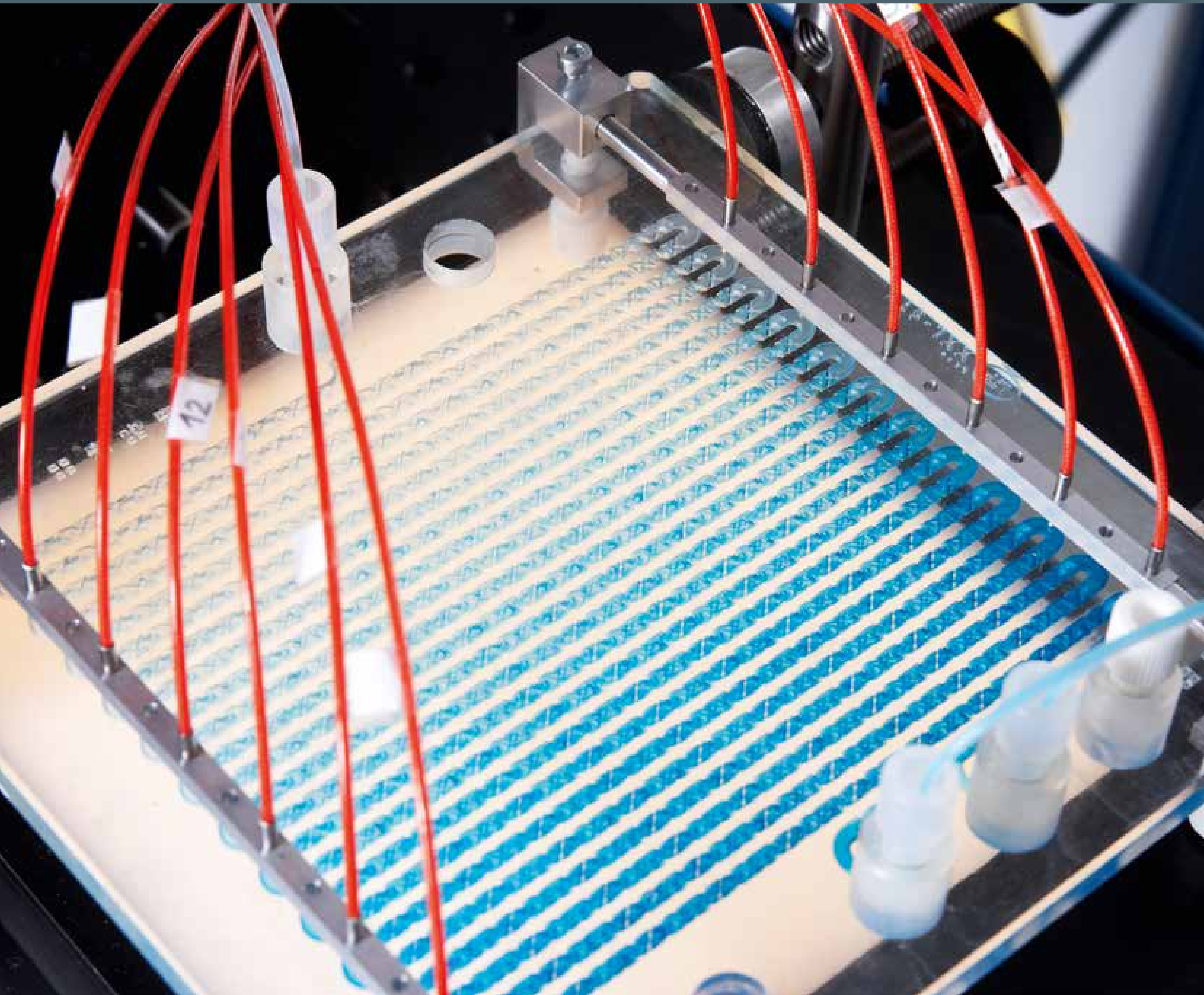
Fraunhofer ICT

FRAUNHOFER INSTITUTE FOR CHEMICAL TECHNOLOGY ICT

PROCESS ANALYTICAL TECHNOLOGY
DIAGNOSIS, OPTIMIZATION AND MONITORING OF CHEMICAL PROCESSES



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DIAGNOSIS, OPTIMIZATION AND MONITORING OF CHEMICAL PROCESSES

Many chemical processes have a significant potential for improvement when the reaction pathways leading to product or byproduct formation and the underlying kinetics are analyzed and understood in detail. The data collected in these analyses can help to optimize processes, ensuring the high and consistent quality of the product and reducing costs.

Spectroscopic process analysis

The trend in the processing industry is increasingly moving away from individual sensors integrated in the process towards innovative process monitoring tools, in particular spectroscopic measurement techniques. At the Fraunhofer ICT, inline and online process analysis is carried out in an early stage of the design and optimization of chemical processes. Vibrational (Raman, mid-infrared and near-infrared) spectroscopy and absorption (UV/Vis) spectroscopy are therefore used individually or combined and adapted to the chemical process. Specially-designed optical measurement cells often serve as a process interface.

Besides process diagnosis and optimization, process analytical data are also used for the active control and automation of chemical processes. This is used both for quality control and for the early detection and counteracting of critical process states, which is particularly relevant to the process safety.

Spectroscopic analysis in microreactor processes

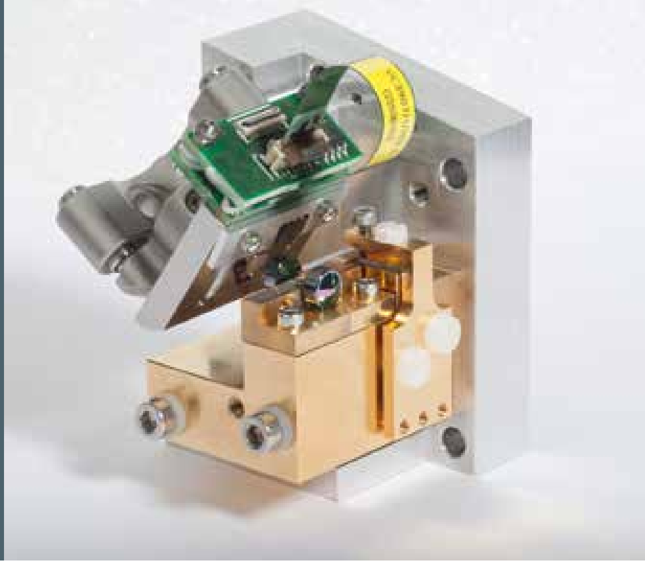
In industrial practice it is often difficult or even impossible to install and implement process analysis on-site, for technical, economic or safety reasons. Continuously operated microreaction technology offers a solution, replicating technical processes on a small scale and monitoring them in high temporal and spatial resolution with the help of adapted process analysis. In the field of process development, microreaction technology allows systematic screening of different process parameters, including some that were previously not accessible. By applying statistical design of experiments and chemometric methods providing quantitative analysis of the product composition in real-time, appropriate process windows and optimal process conditions can be identified.

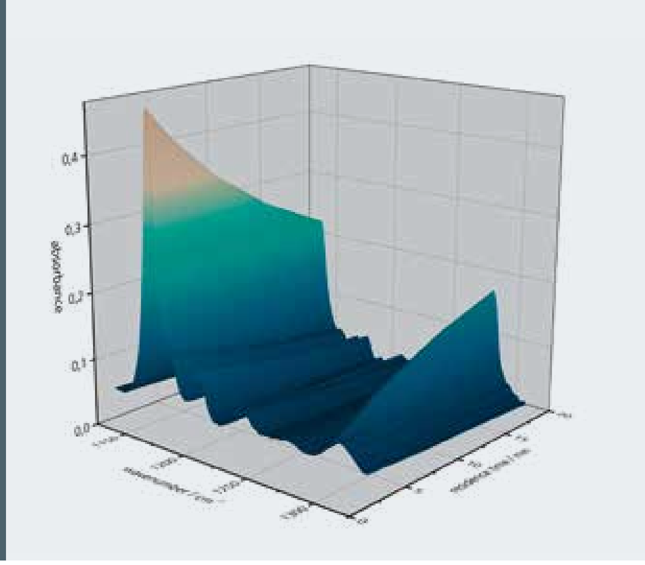
Process analysis with a high temporal and spatial resolution

At the Fraunhofer ICT, modern imaging methods, such as pushbroom imaging, are used to collect spectral data from various measuring points simultaneously. With this method chemical processes can be monitored spectroscopically in real-time with a high spatial resolution in a selected section of a reactor. Using optical fibers, imaging techniques can be applied for multiplex spectroscopy to analyze and monitor continuous processes. This method enables spectroscopic measurements to be carried out at many discrete, freely selectable points, recording the progress of the chemical process over a longer distance. This type of reaction or process tomography provides a multitude of kinetic and mechanistic data. Pushbroom imaging can currently be applied in the UV, VIS and NIR spectral range.

The use of optical fibers now also enables multiplex spectroscopy in the mid-infrared (MIR) range, to track chemical processes at several measuring points. Combined with the high selectivity and specific information content in this spectral region, this makes infrared spectroscopy an important process analysis tool, especially for reaction screening and process design in an early development stage.

SERS analytic module for high-throughput screenings of micro-segmented flow processes.





The latest developments in the field of high temporal resolution spectroscopy in the mid- infrared range are based on the use of new laser-based light sources. The Fraunhofer ICT applies innovative miniaturized quantum cascade lasers (QCL) which are developed in a consortium with other Fraunhofer institutes. The high spectral brightness, fast spectral tuning in the kHz-range, highly compact design as well as the simple process integration of these new semiconductor lasers offer completely new possibilities for the use of infrared spectroscopy in process analysis.

Spectroscopy software

Customized process analytical systems require flexible software solutions. The software RecoMat, developed at the Fraunhofer ICT, enables the control of numerous spectrometers in order to analyze or edit spectra directly, as well as conducting qualitative and quantitative online analyses. Besides pure spectrometer control, aiming for process automation, the software can also be incorporated in a higher-level process and communicate with the control technology using standard protocols. In combination with corresponding actuators, the control system is thus expanded to an automated in-line process control. RecoMat allows both the creation of user-defined spectra pretreatment methods and the integration of user-generated chemometric calibration models for online quantification (prediction) and online identification (classification). With the assistance of multi-channel spectra, the software can also be used in multiplex mode and for spatially highly resolved process spectroscopy, such as pushbroom imaging.

Surface-enhanced Raman spectroscopy

Through the use of surface-enhanced Raman spectroscopy (SERS) the restrictions of conventional Raman spectroscopy, namely the low detection limit and sensitivity, can be overcome. In particular in biological and biochemical screening applications, SERS provides specific information on the material composition and is, in contrast to commonly-used UV/Vis- or fluorescence spectroscopy, label-free. At the Fraunhofer ICT, an at-line SERS analytic module for high-throughput screenings of micro-segmented flow processes was developed. It is based on a SERS-functionalized array on which up to 500 sample droplets of a continuous segmented flow can automatically be detected, deposited and analyzed with a Raman probe.

PICTURES ABOVE

Left: Miniaturized mid-infrared laser source combining a QCL chip in an external cavity (EC-QCL) with a fast scanning optical micro grating as a wavelength selective element (© Fraunhofer IAF, Freiburg).
Right: 3D plot of a reaction sequence monitored by QCL spectroscopy.

PICTURE RIGHT

Multiplex IR-spectroscopy based on optical fibers in a continuous micro-reactor process.

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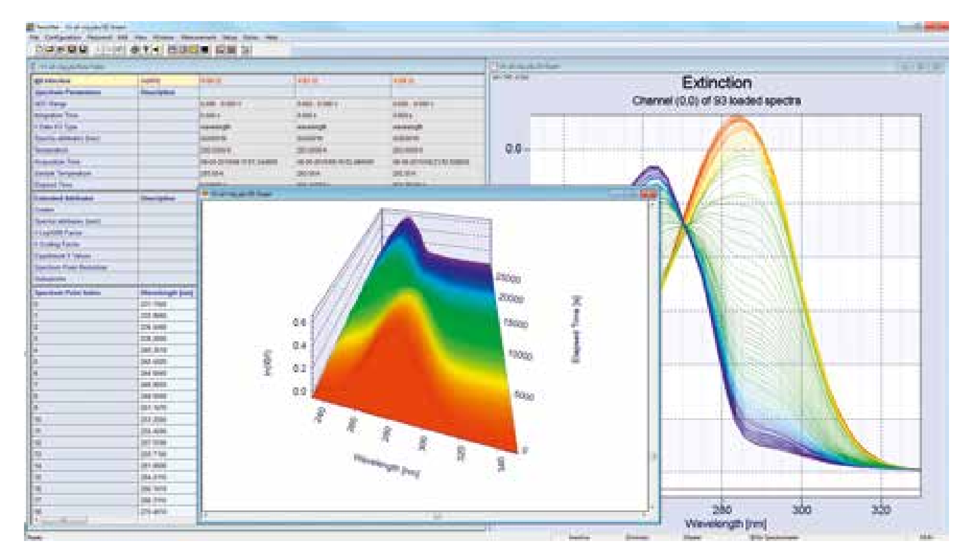

OUR OFFER

The spectroscopic process analysis techniques developed and applied by the Fraunhofer ICT provide insights into the chemical processes of our customers. We collect information on the product composition as well as kinetic and mechanistic data concerning the process in real-time, which are crucial for the design of process components and the selection of adequate process conditions. Combining this with screening methods, statistical design of experiments and chemometric methods, we identify appropriate process windows and optimum process conditions.

On behalf of our customers we conduct feasibility studies, detailed analyses of individual process steps and the design and optimization of chemical processes from laboratory to pilot scale.

PICTURE ABOVE

Graphical user interface of the spectroscopy software RecoMat developed at the Fraunhofer ICT.

PICTURE LEFT

SERS analytic module for high-throughput screenings of micro-segmented flow processes.

COVER PICTURE

Spatially and temporally high-resolution process analysis in a continuous reactor using pushbroom imaging.

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