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"Gh. Asachi" Technical University of Iasi, Romania

Book review

MICRO INSTRUMENTATION
For high throughput experimentation and process intensification – a tool for PAT

K. M. VandenBussche and R.W. Chrisman (Eds)

WILEY-VCH Verlag GmbH&Co. KGaA, Weinheim, Germany
ISBN: 978-3-527-31425-6, 2007, XXIII+495 pags.

Microinstrumentation is o book managed by a team of editors:

Melvin V. Koch is director of the Center for Process Analytical Chemistry (CPAC) and Affiliate Professor of Chemical Engineering at the University of Washington in Seattle.

Kurt M. VandenBussche currently manages the development of UOP's exploratory platform technologies.

Ray W. Chrisman is president of Atodyne Technologies and a visiting scholar at CPAC.

The book reveals the interest in process intensification and capital and operating costs diminishing by miniaturization approaches, which have resulted in the application of micro-instrumentation to the areas of process development and process optimization.

The work includes contribution of many authors of universities and research bodies from USA, Germany and UK.

In the first part *Introducing the Concepts*, some aspects concerning analytical tools for use in Process Analytical Technology (PAT) as well as the topics covered by the Center for Process Analytical Chemistry and Summer Institute are first presented in "Introduction".

This part of the book is organized in four chapters.

Chapter 2 *Macro to Micro… The Evolution of Process Analytical Systems* authors Wayne W. Blaser and Ray W. Chrisman, discusses about the correlation between analytical instruments and chemical process: past technology innovation has had a relevant impact on the evolution of process analytical instrumentation, and new development in the micro-instrumentation area are expected to influence the growth in the field.

A general overview of the current state of analytical instruments and analytical techniques (gas and liquid chromatography, spectroscopy and spectrometry, microflow) is carried out.

Chapter 3, *Process Intensification*, written by Kurt M. VandenBussche introduces the concept of process intensification, giving also some relevant examples from industry.

The author defines process intensification in a broad sense as "a series of methodologies aimed at reducing the capital cost associated with chemical processing by removing existing limitation". Also, he analyses the fields of the process intensification, reaction engineering, gas-phase mass transfer, liquid-liquid mass transfer (mixing and emulsions), gas-liquid mass transfer, mass transfer and gas-solid systems, heat transfer.

The author concluded that the use of miniaturization of equipment can lead to an increase of heat and mass transfer coefficient by several orders of magnitude.

Two case studies illustrate the impact of process intensification (distributed production of methanol and distributed production of hydrogen) looking somewhat ahead to a scenario of multiple energy sources.

Chapter 4, *High Throughput Research*, author Ray Chrisman explores the lessons learned in the early stages of high throughput research for process development, discussing how micro-instrumentation makes this concept much more reasonable, but also exhibiting the current barriers and limitations.

Some concrete aspects are examined: concept of research process, continuous operation and on-line analysis, extracting information from processes, process development, microreactors for process development, microscale reaction characterization.

Part II, *Technology Development and Case Studies*, is structured on 11 chapters, being the most consistent part of the book. It includes studies on the ways to combine effective sampling and sample conditioning with monitoring tools, as elegant means of gathering intrinsic data within microtechnology, combined with proper resources, which could provide direct access to the underlying mechanisms and kinetics of various steps of a certain chemical process. Some examples are provided, which show recent developments and achievements of microtechnology systems for studying and controlling chemical reactions.

Chapter 6, *Microreactor Concepts and Processing*, authors: Volker Hessel, Partick Löb, Holger Löwe, Gunther Kolb introduces and analyses microreactors as a milestone in getting the technology accepted. Also, microreaction technology is considered to deliver novel innovative tools for the chemical processing industry.

Some microstructured systems are examined such as: microstructured mixer-reactors for pilot and production range and scale-out, caterpillar microstructured mixer-reactors, microstructured heat exchanger-reactors, fine-chemical microreactor plants. Also, process development issues are highlighted for industrial production of fine chemicals.

Microreactor laboratory-scale process developments are approached in relation with future industrial use. Also, future directions are revealed considering the potential of microreactors for organic reactions, because they would make possible the overcome of some limitation (mainly kinetic). The conclusion is that microsystems technology advantages, most pronounced at the laboratory scale have to be supplemented by competences in plant and process engineering.

Chapter 7, *Non-reactor Micro-component Development*, authors: Daniel R. Palo, Victoria S. Stenkamp, Jamie D. Holladay, Paul H. Humble, Robert A. Dagle, Kriston P. Brooks, reviews research and development activities related to non-reactive applications of microchannel-process technology, covering heat transfer, mixing, emulsification, phase separation, phase transfer, biological process, body force applications. Ongoing activities in microchannel process technology development from single channel laboratory experiments to industrially-driven, multichannel and multi-unit development are overviewed.

Research results on some chemical unit operations are presented: heat transfer, mixing, emulsification, phase separation, phase transfer, but also results concerning biological processes are evaluated. The conclusion is that microchannel process technology has broad application, even in non-reactive systems.

Chapter 8, *Microcomponent Flow Characterization*, written by a large group of authors, outlines correlations and pertinent theoretical concepts for slow velocity flow and diffusion: pressure drop, entry length, mixing due to connection and diffusion. The work is conducted using computational fluid dynamic approach (CFD) assisted by the program Comsol Multiphysics.

Engineering correlations are presented and analyzed for pressure drop and entry length by considering the common geometrical flow channels and laminar flow. Mixing is also studied in laminar and turbulent flow regimes, even when there are flow and concentration variations. The importance of flow and diffusion characterization using CFD is illustrated in relation with a reaction system.

Chapter 9, *Selected Development in Micro-analytical Technology* describes various technologies that exemplify improvements in analytical field for achieving faster analytical response, when minimized components are involved, selected from the technologies developed and studied at CPAC. These developments include: mixing efficiency, cleaning validation, particles sizing, chemical composition, coating characterization, vapor characterization, viscosity/rheometrics, moisture, bio-assay.

The analysis refers to: application of on-line Raman spectroscopy to characterize and optimize a continuous microreactor, developments in ultra micro gas analyzers, nuclear magnetic resonance stereoscopy, surface plasmon resonance (SPR) sensors, dielectric spectroscopy, liquid-phase microseparation devices in process analytical technology, grating light reflection spectroscopy.

Chapter 10, *New Platform for Sampling and Sensor Initiative (NeSSI),* written by David J. Welthkamp, describes a new type of fluidic system developed by the Chemical and Petro-chemical Industries for process analyzers and related samples handling systems, under the aegidis of NeSSI™ (New Sampling/Sensor Initiative), which is largely presented in the chapter body.

Some details are given about the philosophy and commercial implementation of new sampling systems developed under NeSSI™ program.

Chapter 11, *Catalyst Characterization and Gas Phase Processes*, authors Michelle J. Cohn and Douglas B. Galloway, details some of the tools for kinetics reactivity and diffusion characterization that help to provide a better understanding of catalytic processes and to improve the rate of process development.

Chapter 12, *Integrated Microreactor System for Gas Phase Reactions*, authors David J. Quiram et al., discusses the context developed by microfabrication technology that provides a platform for invention of highly instrumented microscale reactor systems, which incorporate new innovative analytical capabilities, allow point-of-use synthesis and small-scale manufacturing, implementation of novel high-throughput screening methods.

Chapter 13, *Liquid Phase Process Characterization*, authors Daniel A. Hickman and Daniel D. Sobeck, deals with the issues of development of a system devoted to the characterization of homogeneous liquid phase reactions using serial screening in a tubular reactor. The studied system uses a single microchannel reactor with reactants injected as finite pulses. In order to design or select the system components to enable analysis of the reactor effluents at undiluted, steady-state concentrations while injecting only microliters of reactant solutions per experiment, fundamental reactor engineering principles (equation and constraints for heat transfer, mixing, axial dispersion, pressure drop are considered. Also, experimental data that validate the behavior of the reactants are provided.

The tubular reactor was chosen for the study of low axial dispersion of injected reactants, in order to analyze axial dispersion of Newtonian fluids in circular tubes.

Chapter 14, *Novel Systems for New Chemistry Exploration*, author Paul Watts, evidences the performance of microreactors – as a network of micron-sized channels etched into a solid substrate, applied in pharmaceutical industry and fine chemicals synthesis.

Some chemical synthesis in microreactors are described (synthesis of pyrazoles, peptides) as well as reaction optimization, stereochemistry. Chemical synthesis in flow reactors is approached in relation with compounds purification. The conclusion is that reactions performed in microreactors generate relatively pure products in high yield, in comparison with equivalent bulk reactions, and in much shorter time. A very important feature is that the reactants and products are separated in real time, so that rapid screening is facilitated.

Chapter 15, *Going from Laboratory to Plant to Production using Microreactors*, authors: Michael Grund , Michael Harbel, Dick Schmaez, Hanns Wurziger discusses about the unique property of miniaturized reaction systems in the context of production in the multikilogram up to tone scale.

Case studies concerning the application of microreaction technology at Merck KGaA are presented, that include: nitration, microreaction system "MICROTAUROS", automated reaction optimization, upscale in larger laboratory scale, upscale in pilot plants.

The fact that the continuous initiation occurs in a reaction volume diminished by about two orders of magnitude markedly increases the inherent safety, as well as the conversion and selectivity.

Part III, *A Summary and Path Forward contains concluding Remarks* drawn by Melvin V. Koch, Ray W. Chrisman and Kurt M. VanderBussche. A summary of the work is provided, together with the path forward.

References are given throughout the book including basic works as well as papers on the new research and development in the field.

The book written in very modern manner is very useful for chemical and process engineers, chemists, analytical chemists, materials scientists, chemical equipment engineers, specialists in pharmaceutical and fine chemical synthesis.

Maria Gavrilescu
Camelia Bețianu
Florentina Anca Căliman
Department of Environmental Engineering and Management
Technical University of Iasi