

Investigation of runaway reactions using microreaction technology

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Introduction

Runaway reactions always posed a major hazard in the chemical industry and are still dangerous to this day. Companies undertake different investigations to minimize the risk of a thermal runaway.

Having a substantial knowledge of reaction kinetics and thermokinetics offers the basis for the correct design of the chemical reactor and cooling equipment.

Investigations of the runaway reaction are most often focused on the heat flow during the runaway reaction and not its kinetics.

In this work, the aim is to investigate the runaway reaction using the Ehrfeld MMRs (Modular Micro Reactor System) to determine the kinetics of the thermal runaway. This could potentially offer a different but complementary approach to the typically used calorimeter.

Measurement principle



Reactor setup



Fig. 1: Schematic of the reactor setup. IP-001: Inlet pump, IP-002: Micro Heat Exchanger, IP-003: Inlet pump, MV-001: Micro Heat Exchanger, MV-002: Micro Heat Exchanger, MV-003: Micro Heat Exchanger

Integral Reactor

- Ehrfeld Fixed-Bed Meander Reactor
- Fluidic temperature control [-10 – 200°C]
- Used to set the conversion for the differential reactor

In-Line NIR

- Antaris™ II FT-NIR-Analysator
- Used to measure the conversion entering the differential reactor
- Model to fit the conversion to the NIR-spectrum is calculated

Differential Reactor

- Ehrfeld Cartridge Reactor with Fixed-Bed
- Fluidic temperature control [-10 – 200°C]
- Small conversions for the investigation
- Integrated temperature probes to calculate a heat balance

Off-Line Analytics

- GC/MS to analyze the quenched reaction mixture

Installation of the temperature probes

- Installation required 3D printing new modules for the Ehrfeld MMRs
- Modules fit onto pre-existing platforms



Fig. 2: Installation of the temperature probe (3D-model)

Challenges when setting up the reactor

- Installation of the temperature probes (3D printed new module)
- Installation of the in-line NIR probe (used existing module for conductivity sensor)

Reaction Calorimeter

- Produced heat of runaway is measured
- Larger S/V-ratio → less ideal
- Investigation of the whole reaction volume

→ Thermokinetic investigation of the runaway reaction



Fig. 3: Comparison of Reaction Calorimeter and Micro Reactor setup

Vs Micro Reactor setup

- Reaction kinetics of runaway are determined
- Small dimensions → nearly ideal flow and heat regime
- Investigation of a single volume element

→ Kinetic investigation of the runaway reaction

Investigated Reaction

- Reaction between acetic anhydride and methanol, catalyzed by sulfuric acid
- Well documented reaction → used to validate the measurements
- Measurements in the standard region as validation
- Method development in the runaway region



Outlook

- Validating the reactor setup using the standard reaction
- Developing the method for investigation of the runaway reaction
- Validating the temperature probe using the standard reaction

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- [3] DEMOUMET, A., et al., 2012, Risk analysis of French chemical industry. *Safety Science*, 125, pp. 5, 73-85.

Microreaction Technology

Jun-ichi Yoshida



Microreaction Technology:

Microreaction Technology M. Matlosz, W. Ehrfeld, J.P. Baselt, 2012-12-06 IMRET 5 featured more than 80 oral and poster communications covering the entire interdisciplinary field from design production modeling and characterization of microreactor devices to application of microstructured systems for production energy and transportation including many analytical and biological applications A particularly strong topic was the investigation of the potential of microstructuring of reactors and systems components for process intensification Perspectives of combining local in situ data acquisition with appropriate microstructuring of actuators and components within chemical and biological devices were explored in order to enhance process performance and facilitate process control Microreaction Technology: Industrial Prospects W. Ehrfeld, 2012-12-06 Miniaturization has cost and time saving advantages for numerous applications in chemistry pharmacy medicine and biotechnology Additionally microreaction technology offers new solutions for the automobile industry and environmental technology e g fuel cells or mobile sensor systems for on the spot analysis Therefore the 3rd International Conference on Microreaction Technology IMRET 3 is an important forum for creating awareness of the wide variety of the new trends in this up and coming discipline *Micro Reaction Technology in Organic Synthesis* Charlotte Wiles, Paul Watts, 2016-04-19 While continuous processes have found widespread application within chemical production members of the research and development communities have historically favored the centuries old technique of iterative batch reactions With the exception of combinatorial and microwave chemistry little had been done to change the way that synthetic chemists c Microreactor Technology and Process Intensification Yong Wang, Jamelyn D. Holladay, 2005 Microreaction technology with its unprecedented heat and mass transfer advantages as well as uniform residence time and flow pattern is one of the few technologies with potential to develop efficient environmentally benign and compact processes Novel fabrication and processing techniques equipment and operational methods are resulting in spectacular developments that go beyond traditional chemical engineering These new developments promise improvements in process plants and lead to the transformation of our concept of chemical plants into compact safe energy efficient and environmentally sustainable processes Microsystems are now available in many devices for commercial applications including micromixers and microreactors as alternative to batch production in pharmaceutical and fine chemical industry lab on chip devices microensors advanced rapid throughput chemical and catalyst screening tools e g combi distributed or portable power and chemical production distributed heating and cooling and even out of this world applications with NASA A wide diversity of subjects are discussed in this book ranging from catalysis to fuel processing to combinatorial techniques to separations to novel reactors all of which are enabled by microtechnology principles World renowned pioneers Klavs Jensen Volker Hessel Jennifer Holmgren and Galip Akay provide accounts on both historical developments and the current state of the art as well as insights into future research and development in microreactor and process intensification Research and developments are

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Micro Process Engineering Norbert Kockmann, 2013-03-26 This edition of Micro Process Engineering was originally published in the successful series Advanced Micro Nanosystems Authors from leading industrial players and research institutions present a concise and didactical introduction to Micro Process Engineering the combination of microtechnology and process engineering into a most promising and powerful tool for revolutionizing chemical processes and industrial mass production of bulk materials fine chemicals pharmaceuticals and many other products The book takes the readers from the fundamentals of engineering methods transport processes and fluid dynamics to device conception simulation and modelling control interfaces and issues of modularity and compatibility Fabrication strategies and techniques are examined next focused on the fabrication of suitable microcomponents from various materials such as metals polymers silicon ceramics and glass The book concludes with actual applications and operational aspects of micro process systems giving broad coverage to industrial efforts in America Europe and Asia as well as laboratory equipment and education

Re-Engineering the Chemical Processing Plant Andrzej Stankiewicz, Jacob A. Moulijn, 2018-12-14 The first guide to compile current research and frontline developments in the science of process intensification PI Re Engineering the Chemical Processing Plant illustrates the design integration and application of PI principles and structures for the development and optimization of chemical and industrial plants This volume updates professionals on emerging PI equipment and methodologies to promote technological advances and operational efficacy in chemical biochemical and engineering environments and presents clear examples illustrating the implementation and application of specific process intensifying equipment and methods in various commercial arenas

Microreactors in Preparative Chemistry Wladimir Reschetilowski, 2013-09-13 This is the first book in the field to focus on these aspects providing extremely valuable information unavailable elsewhere for anyone seeking the practical application of microreactor technology in preparative chemistry The topics covered branch out in three different directions To begin with the knowledge necessary for the preparative chemistry concerning the influence of the so called microeffects on the reaction procedure and on mass and heat transfer as well as the surface phenomena are provided in detail Next practical aspects of the synthesis of various basic chemicals and fine chemicals polymers bioproducts and nanoparticles are discussed including important advice for both the researcher and industrial chemist Finally reaction examples in microreactors whose reaction guidance are best understood are given together with universally applicable correlations as well as modeling approaches and transfer potential on related reaction systems With its specific instructions tips and experimental procedures for product

syntheses as well as the inclusion of both the technical and theoretical background this is a must have for beginners and experts alike working in this emerging field

Electrochemical Microsystem Technologies J. Walter Schultze, Tetsuya Osaka, Madhav Datta, 2002-09-05 Driven by the electronics industry electrochemical technology has rapidly evolved finding increasing applications in microelectronics batteries sensors materials science industrial fabrication corrosion microbiology neurobiology and medicine Electrochemical Microsystem Technologies provides an overview of the technological status the dev

Chemical Micro Process Engineering Volker Hessel, Steffen Hardt, Holger Löwe, 2006-03-06 Micro process engineering is approaching both academia and industry With the provision of micro devices and systems by commercial suppliers one main barrier for using these units has been eliminated More and more they become familiar thereby being one facet of the upheaval in chemical industry This book focuses on processes rather than on devices what is before and behind micro device fabrication A comprehensive and detailed overview is given on A multi faceted hierarchic analysis of chemical micro process technology Modelling and simulation of micro reactors Liquid and liquid liquid phase reactions Gas liquid reactions Gas phase reactions heterogeneous catalysis

Microreactors for Gas/Liquid Reactions Kristin Hecht, 2014-01-01 The widespread adoption of microreactors has been hindered by a lack of knowledge regarding rules and parameters needed for their design This dissertation deals with the investigation of interfacial area and liquid mass transport in microreactors for gas liquid reactions Observations of a reduction in conversion occurring when changing from a glass laboratory device to a stainless steel process device motivated investigations into the effect of material and surface properties on the gas liquid flow and reaction This work takes a look inside the machined microstructures produced at the Institute for Micro Process Engineering of the Karlsruhe Institute of Technology and investigates the nature of the solid surfaces resulting from the machining process the surface roughness and the contact angle of the resulting surface The effect of wettability on gas liquid flow in microreactors has been investigated in capillaries of various materials The absorption of carbon dioxide into aqueous solutions of sodium hydroxide has been used to investigate gas liquid flow and reaction in capillaries and in a microstructured falling film reactor Under certain limiting assumptions depending upon the exact rates of reaction and transport values of the interfacial area and liquid mass transport coefficient can be derived from measurements of reaction This dissertation contributes a small step towards a better understanding of how measurements of the interfacial area and mass transfer coefficient can be performed demonstrates why the effect of material wettability cannot be ignored and provides a stronger basis for further work in the development of general correlations describing microstructured gas liquid reactors

Microreactors in Organic Synthesis and Catalysis Thomas Wirth, 2008-05-05 This one stop reference is the first book on this emerging and rapid developing field with a focus on synthesis and catalysis As such it covers all aspects from academia and industry in a clearly structured way Leading experts provide the background information as an initial aid for newcomers to the field while chapters on different reaction types and industrial applications

make this an equally vital resource for specialists From the contents Introduction and background Fabrication of microractors Properties and use of microreactors Organic chemistry in microreactors Homogeneous reactions including photochemical and electrochemical reactions Heterogeneous reactions Biphasic reactions liquid liquid liquid gas Bioorganic reactions Industrial applications Thomas Wirth is Professor of Organic Chemistry at Cardiff University in Wales After a postdoctoral stay with Kaoru Fuji at Kyoto University as a JSPS fellow he started his independent research in the group of Bernd Giese in Basel Switzerland He was invited as a visiting professor to various places University of Toronto Canada 1999 Chuo University in Tokyo Japan 2000 Osaka University Japan 2004 He was awarded the Werner Prize from the New Swiss Chemical Society in 2000 He is the author of about 80 publications and has written or edited 4 books

Microreactors in Organic Chemistry and Catalysis Thomas Wirth, 2013-02-22 For the second edition of *Microreactors in Organic Chemistry and Catalysis* all chapters have been revised and updated to reflect the latest developments in this rapidly developing field This new edition has 60% more content and it remains a comprehensive publication covering most aspects of the topic The use of microreactors in homogeneous heterogeneous as well as biphasic reactions is covered in the main part of the book together with catalytic bioorganic and automation approaches The initial chapters also provide a solid physical chemistry background on fluidics in microdevices Finally a chapter on industrial applications and developments covers recent progress in process chemistry An excellent reference for beginners and experts alike

Basic Principles in Applied Catalysis Manfred Baerns, 2013-03-09 Applied catalysis is based nowadays not only on empirical knowledge but also on the many insights that have been gained from the fundamental understanding of catalysis It also comprises knowledge and expertise from catalytic reaction engineering in particular kinetics of the catalytic reaction and its interplay with heat and mass transfer as well as fluid dynamics and the specific conditions prevailing in the type of reactor used Applied catalysis comprises many areas from a reaction point of view many types of catalytic materials from which catalysts are formed are needed to achieve high selectivities and space time yields last but not least catalysts should have a long life time to which its deactivation is detrimental A catalytic material that fulfils all the demands then often requires special mechanical and thermal treatment to be used in practise Various books have been written about specific areas as mentioned above It is the intention of this contribution to present timely reports by well recognised experts in the field to outline the state of science and technology in selected but representative areas illustrating the basic principles of applied catalysis

Transport Phenomena in Micro Process Engineering Norbert Kockmann, 2007-11-12 In this book the fundamentals of chemical engineering are presented aiming to applications in micro system technology microfluidics and transport processes within microstructures After a general overview on both disciplines and common areas recent projects are shortly presented The combination of different disciplines gives new opportunities in microfluidic devices and process intensification respectively Special features of the book are the state of the art in micro process engineering a detailed treatment of transport phenomena for engineers a

design methodology from transport effects to economic considerations a detailed treatment of chemical reaction in continuous flow microstructured reactors an engineering methodology to treat complex processes The book addresses researchers and graduate students in the field of chemical engineering Microsystems engineering and chemistry Green Separation Processes Carlos A. M. Afonso, João Pedro G. Crespo, 2006-05-12 This timely book is the first to provide a comprehensive overview of all important aspects of this modern technology with the focus on the green aspect The expert authors present everything from reactions without solvents to nanostructures for separation methods from combinatorial chemistry on solid phase to dendrimers The result is a ready reference packed full of valuable facts on the latest developments in the field high quality information otherwise widely spread throughout articles and reviews From the contents Green chemistry for sustainable development New synthetic methodologies and the demand for adequate separation processes New developments in separation processes Future trends and needs It is a must have for every researcher in the field

Modeling of Process Intensification Frerich J. Keil, 2007-06-27 Combining the knowledge involved in process engineering and process modeling this is the first book to cover all modeling methods applicable to process intensification Both the editors and authors are renowned experts from industry and academia in the various fields of process modeling and integrated chemical processes Following an introduction to the topic the book goes on to look at equipment and operational methods monolithic catalysis HEX micro and reverse flow reactors catalytic and reactive distillation the simulated moving bed and vibration bubble column as well as ultrasound and ultrasonic reactors A final chapter is devoted to processes under supercritical conditions In its treatment of hot topics of multidisciplinary interest this book is of great value to researchers and engineers alike

Micro Process Engineering, 3 Volume Set Volker Hessel, Albert Renken, Jaap C. Schouten, Jun-Ichi Yoshida, 2009-03-23 This three volume handbook provides an overview of the key aspects of micro process engineering Volume 1 covers the fundamentals operations and catalysts volume 2 examines devices reactions and applications with volume 3 rounding off the trilogy with system process and plant engineering Fluid dynamics mixing heat mass transfer purification and separation microstructured devices and microstructured reactors are explained in the first volume Volume 2 segments microreactor design fabrication and assembly bulk and fine chemistry polymerisation fuel processing and functional materials into understandable parts The final volume of the handbook addresses microreactor systems design and scale up sensing analysis and control chemical process engineering economic and eco efficiency analyses as well as microreactor plant case studies in one book Together this 3 volume handbook explains the science behind micro process engineering to the scale up and their real life industrial applications

New Developments and Application in Chemical Reaction Engineering Hyun-Ku Rhee, In-Sik Nam, Jong Moon Park, 2006-05-10 This Proceedings of APCRE 05 contains the articles that were presented at the 4th Asia Pacific Chemical Reaction Engineering Symposium APCRE 05 held at Gyeongju Korea between June 12 and June 15 2005 with a theme of New Opportunities of Chemical Reaction Engineering

in Asia Pacific Region Following the tradition of APCRE Symposia and ISCRE the scientific program encompassed a wide spectrum of topics including not only the traditional areas but also the emerging fields of chemical reaction engineering into which the chemical reaction engineers have successfully spearheaded and made significant contributions in recent years In addition to the 190 papers being accepted six plenary lectures and 11 invited lectures are placed in two separate chapters in the front Provides an overview of new developments and application in chemical reaction engineering Topics include traditional and emerging fields Papers reviewed by experts in the field

Basics of Flow Microreactor Synthesis Jun-ichi Yoshida, 2015-05-08 This book provides in a concise form the principles and applications of flow microreactors in organic and polymer synthesis Recently it became possible to conduct chemical reactions in a flow reactor in laboratory synthesis The flow microreactor enables reactions that cannot be done in batch opening a new possibility of chemical synthesis Extremely fast mass and heat transfer and high resolution residence time control are responsible for the remarkable features of that process The book is not an exhaustive compilation of all known examples of flow microreactor synthesis Rather it is a sampling of sufficient variety to illustrate the concept the scope and the current state of flow microreactor synthesis Researchers both in academia and in industry will be interested in this book because the topics encompassed by the book are vigorously studied in many university and company laboratories today

Microstructured Devices for Chemical Processing Madhvanand N. Kashid, Albert Renken, Liubov Kiwi-Minsker, 2014-12-22 Faster cheaper and environmentally friendly these are the criteria for designing new reactions and this is the challenge faced by many chemical engineers today Based on courses taught by the authors this advanced textbook discusses opportunities for carrying out reactions on an industrial level in a technically controllable sustainable costeffective and safe manner Adopting a practical approach it describes how miniaturized devices mixers reactors heat exchangers and separators are used successfully for process intensification focusing on the engineering aspects of microstructured devices such as their design and main characteristics for homogeneous and multiphase reactions It addresses the conditions under which microstructured devices are beneficial how they should be designed and how such devices can be integrated in an existing chemical process Case studies show how the knowledge gained can be applied for particular processes The textbook is essential for master and doctoral students as well as for professional chemists and chemical engineers working in this area

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