Project Overview

Combining Process Intensification-driven Manufacture of Microstructured Reactors and Process Design regarding to Industrial Dimensions and Environment

FP7-NMP-2008-LARGE-2
Process intensification vs. optimization

- Concepts and objectives
  - Topics addressed in the call
  - Problem situation
    - Main objectives and breakthroughs
    - Concepts
- Progress beyond the state-of-the-art
- S/T methodology
- Expected impacts
- Dissemination and exploitation
## Process intensification vs. optimization

<table>
<thead>
<tr>
<th>Aim</th>
<th>Process optimization</th>
<th>Process Systems Engineering</th>
<th>Process intensification</th>
</tr>
</thead>
<tbody>
<tr>
<td>Focus</td>
<td>Performance improvement of existing concepts</td>
<td>Multi-scale integration of existing/new concepts</td>
<td>Development of new concepts of process steps &amp; equipment</td>
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<tr>
<td>Focus</td>
<td>Model, numerical method</td>
<td>Model, software</td>
<td>Experiment, phenomenon, interphase</td>
</tr>
<tr>
<td>Interdisciplinarity</td>
<td>Weak, Applied mathematics</td>
<td>Modest, Applied mathematics &amp; informatics, chemistry</td>
<td>Strong, Chemistry &amp; catalysis, applied physics, mechanical engineering, materials science, electronics, etc.</td>
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### Top-10 Patentees

<table>
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<tr>
<th>Rank</th>
<th>Patent Publications</th>
<th>Patentee</th>
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<tr>
<td>1</td>
<td>144</td>
<td>MERCK PATENT GMBH</td>
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<tr>
<td>2</td>
<td>135</td>
<td>BATTELLE MEMORIAL INSTITUTE</td>
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<td>3</td>
<td>129</td>
<td>VELOCYS INC.</td>
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<td>4</td>
<td>54</td>
<td>FORSCHUNGSZENTRUM KARLSRUHE GMBH</td>
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<td>5</td>
<td>50</td>
<td>UHDE GMBH</td>
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<tr>
<td>6</td>
<td>46</td>
<td>SIEMENS AKTIENGESELLSCHAFT</td>
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<td>7</td>
<td>45</td>
<td>CASIO COMPUTER CO., LTD.</td>
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<td>8</td>
<td>40</td>
<td>DEGUSSA AG</td>
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<td>9</td>
<td>39</td>
<td>BAYER AG</td>
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<tr>
<td>9</td>
<td>39</td>
<td>INSTITUT FUER MIKROTECHNIK MAINZ GMBH</td>
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<tr>
<td>10</td>
<td>37</td>
<td>CLARIANT INTERNATIONAL LTD.</td>
</tr>
</tbody>
</table>

**Megatrends**

- Nanotechnology
- Lab on a Chip
- Microreactors
- Micromixers
- Microactuators

**Nation wise**

![Graph showing patent publications by country over years](image)

<table>
<thead>
<tr>
<th>State-of-the-art</th>
<th>Progress beyond – delivery item of this project</th>
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<tbody>
<tr>
<td>Wash-coat catalysts and similar medium-porous materials</td>
<td>Introduction of highly porous hierarchic artificial catalyst structures to microchannels</td>
</tr>
<tr>
<td>Multipurpose reactor is needed</td>
<td>Unified plate-type microstructured reactor design. Modular platform with all utilities in compact format</td>
</tr>
<tr>
<td>80% of equipment on market is not (yet?) suitable, only useful for product development</td>
<td>New, mass-suited microfabrication techniques (for making large plates or multiple small plates)</td>
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<tr>
<td>Solutions for downstream processing, in combination with PI reactors, hardly exist</td>
<td>Specific downstream processing solutions for continuous operation (by DEG) in fine chemistry; downstream cabinets in pilot plant container</td>
</tr>
<tr>
<td>No pilot facilities or possibilities to pilot on existing production lines</td>
<td>Development of mini-production plant, tailored for PI and micro process technology investigations. Mobile pilot facility</td>
</tr>
<tr>
<td>No integrated intensified multi-product plant is available which provides short shut-down, start-up and change-over times, process sustainability (capital-operating costs) safety, assurance and product quality</td>
<td>Ammonia production from biomass via gasification will provide a model plant which can be retrofitted with additional reactors to produce higher chemicals from ammonia or enhance efficiency.</td>
</tr>
<tr>
<td>Retrofitting PI technologies. In current plants difficult</td>
<td>Retrofit in syngas-ammonia plant</td>
</tr>
<tr>
<td>High (technical and financial) risk of development of first industrial prototype and of first implementation (retrofitting) of PI</td>
<td>Novel Process Windows enable highly intensified and cost-effective routes. Ex-ante cost studies</td>
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<tr>
<td>Reliability (non proven technology)</td>
<td>Six demonstration cases at industrial site to show up reliability for one pilot platform</td>
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</table>
## Costs and soft human factor

### State-of-the-art | Progress beyond – delivery item of this project
--- | ---
Insufficient pay-back on New process technology | Ex-ante cost analysis
Long development path | Mini-production plants to be developed in flexible functional-module or cell-based container format
Suppliers for industrial applications are lacking - most novel equipment only suitable for product development | Build-up of supply chains: Industrial machining manufacturers

### State-of-the-art | Delivery item of this project | Beyond the scope of this project
--- | --- | ---
Lack of PI knowledge and know-how among process technologists | Share of PI know-how and technology from world-leaders to non-practitioners | Not part of project, but need separate investigation
Lack of chain vision | IP cooperation during R&D by patentable plant design is central issue of COPIRIDE | Needs holistic view & analysis of many developments
Insufficient valorization of sustainability | Training and education | Needs long-lasting consistent developments
Paradigm shift, cultural change necessary | One pilot platform facilitates learning | Needs long-lasting education and training efforts
Long learning curve | | Information spread is task beyond project level
Insufficient awareness of potential benefits of PI technologies at the management level | | New generation of decision-makers

### Enthusiasm, frustration, proliferation

- **Market volume for Lab Chips in 2002:** ~ 80 Mio USD
  - **Source:** Yole Development
- **Frost&Sullivan market study 2003:** "Microfluidics will be big!"
  - **Prognosed market volume for 2008:** ~ 700 Mio USD

### Market volume trends:

- **1996**
  - Nexus market study
- **2000**
- **2005**

**Examples of classical applications and areas of new developments within the chemical and process engineering disciplines**

(Source: Chemical Engineering Progress, January 2003, p. 313)
NMP-2008-3.2-1 Implementation of process intensification strategies in industrial scale

Technical content/scope:

• Competition from emerging countries where large quantity production is cheaper and sometimes even more flexible

• Changing customer needs: higher product diversification and more fast and flexible future production strategies

• New concepts for holistic product and process development
  ➢ Flexible integration of inherently safe process technologies
  ➢ New, intensified process and plant concepts for speeding up market penetration, for enhancing the product life-cycle and improving sustainable production

Specific features:

Active participation of industrial partners

(i) exploitation and dissemination, industrial leadership, demonstration activities

(ii) existing activities and initiatives in process intensification are taken into account

(iii) Pharmaceutical active ingredients are not considered a priority
Main development tasks

- Develop new production concepts, new start-up and shut-down strategies
- Exploit the full potential of micro process technologies, integration of different unit operations in one apparatus and standardisation of such modules
- Create flexible systems, adaptable to the dynamic range of product output
- Minimize the use of resources and improve the eco-efficiency
- Create a methodology based on framework data from model reactions, which enables a profound economic as well as technical evaluation of the new production processes and use these methodology to identify transfer-potential of the new processing concepts for innovative products

Project objectives

- Construction and operation of flexible demonstration plants
- Integrated process units and combined unit operations must be linked with process modeling tools, in-line monitoring, model-based process management and advanced process control
- Highly integrated technology demonstration platform for future factory concepts
State-of-the-art … what we need to improve

Reactors
- Microstructured reactors available for laboratory based pilot plants.
- Production-targeting microreactor designs with extended outer dimension, high degree of numbering-up and system integration are rare.

Plants
- Several demonstration runs in dedicated, home-made pilot plants built in industrial laboratories using stock lab equipment.
- Examples to bridge to production in heavy pilots.

Processes
- Process intensification demonstrated for the stage of reaction.
- Missing is a holistic view, taking into account the whole process development cycle.
- Identify additional PI drivers for the whole development chains and to add the respective developments.
Five main innovation drivers

- CATALYSTS
- FABRICATION
- PROCESSES
- REACTORS
- PLANTS

KHCO₃ (aq) → COOH
Micro Process Engineering
A Comprehensive Handbook

Volker Hessel, Jaap C. Schouten, Albert Renken and Jun-Ichi Yoshida (Eds.)

WILEY-VCH


Systemic approach

Fuel Processing

Scale-out devices

Pilot scale

Gas-liquid Falling-film technology

Test devices

Catalysts & Microfluidics

Laboratory scale

Industrial up to World scale

Processes & Plants

Bulk Chemicals

Systems

Novel Process Windows
Upfront cost and LCA analysis
**Main objectives – ‘breakthroughs’**

### NEW, IP PROTECTED MODULAR PRODUCTION & FACTORY CONCEPT
- to provide a *modular plant platform* in block format as flexible functional modules with extension to a mobile and compact cell-based container format, with the surplus of *intellectual property protection* (‘Copyright’ - IP cooperation during R&D)
- to provide a *dedicated integrated plant* being boosted by a 2\(^{nd}\) PI field.
- to fully exploit the *dimorphicity of reactor/plant & process design* by utilizing new modes of operation (*Novel Process Windows*).

### ADAPTABLE PLANTS WITH FLEXIBLE OUTPUT
- to develop an *upgradeable cell format* for the plants allowing to perform laboratory, pilot, and production-scale experiment.

### PRODUCTION-SCALE & MASS-MANUFACTURED MICROSTRUCTURED REACTORS
- to make standardised *microstructured reactor design* accessible for production characterised by large plate extension, large plate numbers, and large numbers of reactors
- to *ground supply chains* through established European companies, being leaders in their branches for the main development lines of the project – machining, reactors, plants; especially for offer of professional products and service at a pilot level.

### INTEGRATED REACTORS AND PROCESSES
- to extend established integrated microstructured reactors (mixer-heat exchanger) to pilot and production format and to develop new ways of integration (membranes, 2\(^{nd}\) PI field).

### REDUCTION OF RESOURCES AND COSTS & IMPROVEMENT OF ECO-EFFICIENCY
- to orient and re-direct developments by commitment to shortcut *ex-ante cost and eco-efficiency analyses* for warranting economic competitiveness and benign, sustainable processing and products

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**Expected impact:**

- **Product diversification**
- Substantially shorter time to process/market
- Flexible production capacity
- Substantial drop in capital expenditure for high value-added product market (< 100,000 t/yr)
## Partners and collaborators

<table>
<thead>
<tr>
<th>Beneficiary Number *</th>
<th>Beneficiary name</th>
<th>Beneficiary short name</th>
<th>Country</th>
<th>Date enter project**</th>
<th>Date exit project**</th>
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<tr>
<td>1(Coordinator)</td>
<td>Institut für Mikrotechnik Mainz GmbH</td>
<td>IMM</td>
<td>DE</td>
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<td>9</td>
<td>Dipartimento di Chimica dell’Università degli studi di Napoli FEDERICO II</td>
<td>UNA</td>
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<td>Friedrich-Schiller-Universität Jena</td>
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</table>

‘Copi riders’

Easy Rider, 40 years ago, Dec. 1969
Partners’ locations and plant sites

Evonik Degussa, Hanau
Evonik Degussa, Rhenfelden
ITI Energy, Rotherham
Mythen, Ferrandina
CHEMEX, Tortona

Partners’ locations and plant sites
Concretion of objectives – real-life delivery items

1. Introduction of highly porous hierarchic artificial catalyst structures to microchannels
2. Expansion, standardisation, and modularisation of microstructured and PI reactor toolbox
3. New, mass-suited microfabrication techniques
4. Modular mini-production plant platforms as frames for the fine-chemical processes
• Modern and highly efficient process- and plant concept covering the whole manufacturing chain to the chemical product in one system – including reactant, product, side product supply and storage, reaction, and purification.

• Several cabinets for logistics, reaction/purification, and instrumentation and control with separate placement of the process control and E-technique units.

- Highly efficient solutions for safety concept, for warranty of on-the-job and environmental safety standards, for process control, in utility equipment, in construction, in the mounting concept, in the reactant & product logistics, cross compliance, and maintenance.
> Supports for standardisation of components and modules.

> Short erection time and a drastic cost reduction.

> Ideally suited for apparatus for process intensification - in particular milli and micro process technological apparatus.

> Decrease in plant footprint and lower investment costs. the innovation cycle can be shortened, advantages in terms of changeover costs and time and purification of the plant for new campaigns.

> Link research with process development, production and plant engineering.

> Upgraded to a mini-production plant of a capacity of a few 100 t/a.

> High flexibility concerning diverse chemical syntheses and correspondingly diverse products.

> Dedicated manufacture of a product leads to lower specific operating costs, since the process can be tailored to the corresponding process.
Concretion of objectives – real-life delivery items (2)

Dedicated plants

Hybrid integrated mini-production plant for ammonia synthesis
Modular platforms have potential for IPR


Shifting reactions to suit Microreactors

Reactions can be designed to suit microreactor technology:

- Increased concentration
  - Harsher reaction conditions
  - Fast reactions
- Increased temperature
- Adjusted residence time RT
- Hazardous reagents and intermediates
- Expensive, but more effective solvent and catalysts
- Controlled educt quality, filtering, avoid impurities and particles

- **Type A reactions**: very fast, < 1 s; mixing controlled
- **Type B reactions**: rapid, 1 s to 10 min; kinetically controlled
- **Type C reactions**: slow, > 10 min; safety and quality issues
Flash chemistry


- CET Special Issue „Novel Process Windows“ November 2009
- International DBU Workshop „Novel Process Windows“; December 10, 2009
Novel Process Windows

Heterogeneous catalytic routes
- Routes bridged by intermediates
- One flow ('pot') multi-step route
- Direct one-step synthesis

Alternative heating (MW)
- Pressurized ex-reflux processes
- Ex-cryogenic processes

Routes at much elevated temperature
New chemical transformations

Routes at much elevated pressure

Routes in the explosive or thermal runaway regime

Process integration and simplification

Mixing all-at-once
- Catalyst-free
- Reduced process expenditure

NOVEL PROCESS WINDOWS

Hazardous reactants
- Thermal runaway regime
- Ex regime

Solvent-free
- Solvent-less
- Alternative solvents (IL, SCF)
Instructive example for NPW guided PI


**Batch (1 l)**
2 h – 7200 s
20 kg/(m³ h)
1 t / a

4 t / a
64200 kg/(m³ h)
4 s
Flow chem (9 ml)

→ Reaction time reduction at best up to 2000 times; increase in space-time yield by factor 3200

![Diagram with chemical reactions and yield percentages]
New products via new process windows

- OLED materials
- Phenols by Kolbe-Schmitt synthesis
- Chitosan for Pharmaceuticals
- High pressure amination of hydrocarbons
- New ink materials

guided and accompanied by LCA & cost analyses

Universities in co-operation with industry:

- Jenpolymers Ltd
- Sigma-Aldrich
- Heppe Medical Chitosan GmbH,
- Pelikan PBS GmbH &CoKG
- ASD GmbH, JTT

+ KONAROM, Zuckerschmelzen, Prof. König
+ CORA, $\text{H}_2\text{O}_2$ via Membranen, Prof. Dittmeyer
There is not one commonly agreed definition on process intensification, but rather relies on a number of existing process parameters, there are 10 rules of green chemistry, there are 1000 criteria for company-internal process evaluation.

Look for a more generalised evaluation procedure in COPIRIDE; one which is not only theoretically sound, but convincing to experts and appealing to decision-makers.
‘Early-bird measuring rods’ – do not decide too late

(Simplified) Life-Cycle Analysis

“Do not lock the stable door after the horse has bolted”

Check for competitiveness

“Be holistic”

Application of decision support and optimisation tools

“Early bird” – ex-ante

Freedom of choice

R&D Scale-up Production

Stage of development

Knowledge
Determination of costs and environmental impacts

Increase of extent and detail within the assessment

Degree of freedom - processing


Facts, facts, facts – ex-ante and how to bundle these for guidance

Multi Criteria Decision Support

Multiobjective Optimisation (MO)

Industrial pilot activities demonstrated by medium and small companies

- Sugar oxidation hydrogenation (Abo Akademi Uni)
- Epoxidation (Mythen)
- Biodiesel production (Chemtex)
- Ammonia production (ITI Energy)
- Polymer chemistry reaction 1 (Evonik-Degussa)
- Polymer chemistry reaction 2 (Evonik-Degussa)
The ultimate ambition of the research to be performed within the project COPIRIDE is:

**To transfer Process Intensification Technology and Know-How from European leaders to European Countries (and respective industries) with no exposure so far, including a transfer from large to SME companies and from chemical to other (food and biofuels) branches.**
Training and Education

Organisation Projektcluster

KARMI-KF

- gemeinsam mit:
  - Nord-Verbund, Dr. Schlüter
  - Niedersachsen, Prof. Turek
  - Brandenburg, Prof. Löhmannsröben
  - Thüringen-Sachsen, Prof. Klemm
  - München, Prof. Lercher

Albert-Ludwigs-Universität Freiburg
Joint workshop presentation of F3 FACTORY, COPIRIDE, PILLS (+ POLYCAT + SYNFLOW)