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Microreaction Technology

Microreaction technology is gaining increasing importance in chemical and pharmaceutical synthesis, chemical engineering and process technology.

What is the advantage of Microreaction Technology?

1) Optimal mixing

- Laminar Flow
- Fast diffusion
- Optimal mixing within seconds

2) Effective heat exchange

- High surface to volume ratio
- Resulting in a good heat transfer
- Heat transfer properties magnitudes better compared to normal equipment.

3) Small reaction volume

- Reaction volume in the range of millimeter
- Allows handling of dangerous reactions, e.g. with explosives or toxic components
- Exact control of reactions and therefore suppression of unwanted side products.

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4) Faster development
    - By fast optimization
    - By numbering up instead of scale up, they can be applied to synthesize kg- or even ton- amount, particularly in parallelized arrays.

What are microreactors?

Microreactors are defined as miniature housings for carrying out chemical reactions.

They have to perform three basic function:

- Initiate and facilitate a reaction through mixing of the reactants,
- Provide time and volume to allow the reaction to finish (residence times),
- To provide or remove heat (heat exchange).

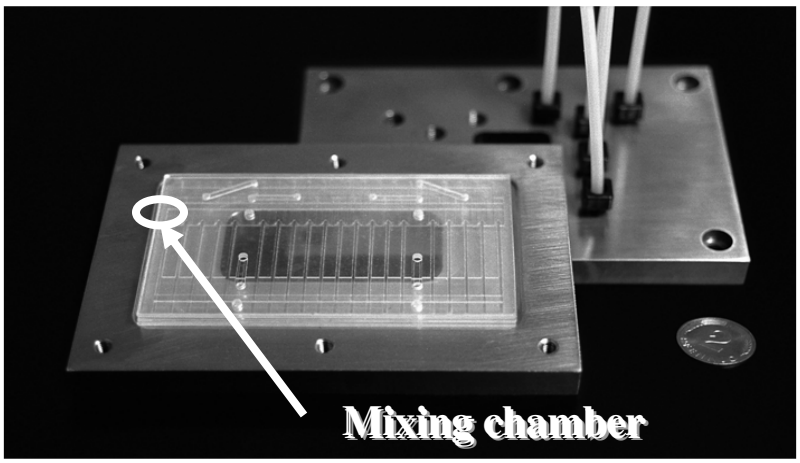

Figure 1. Microreactor.

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mikroglas

Developed in co-operation with Fraunhofer Institut Chemische Technologie'

- exothermal reaction
- cooling down to - 40°C

Channel:

- width 325 µm / 700 µm
- depth 200 µm / 700 µm

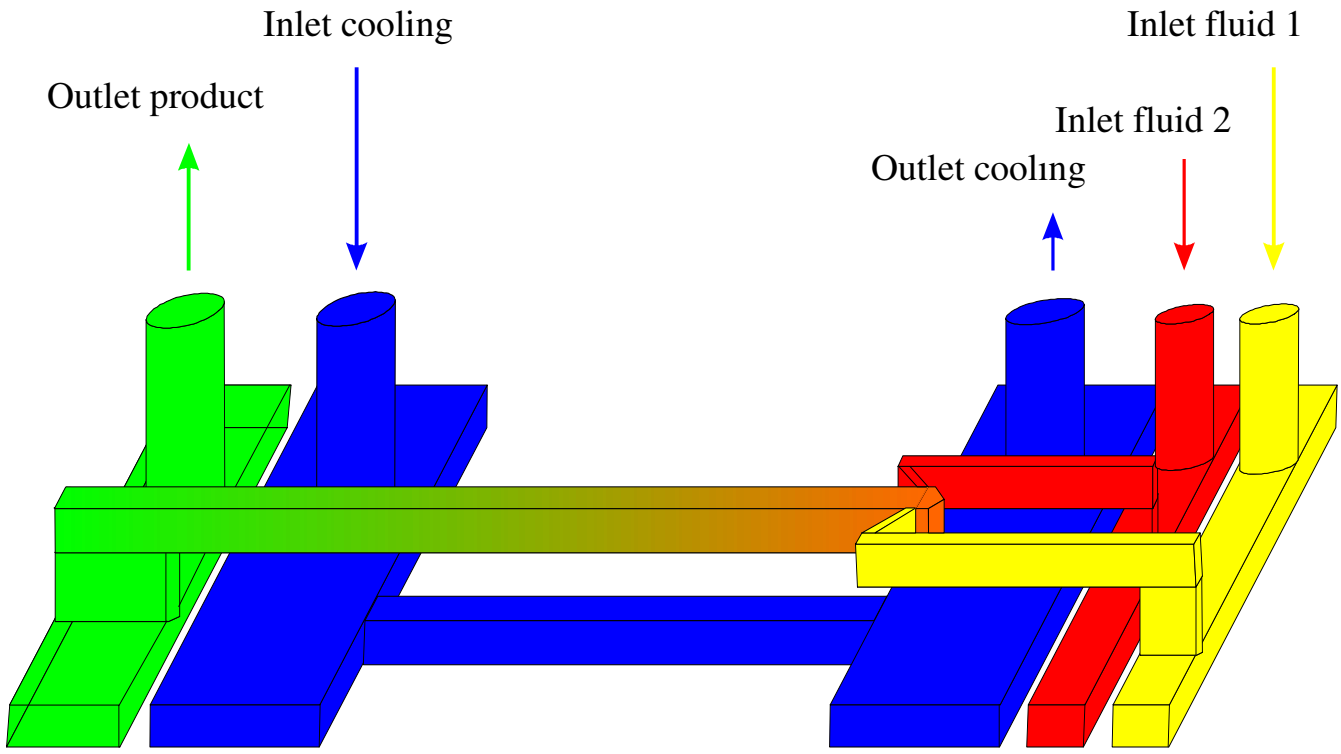

Figure 2: Microreactor – sketch

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mikroglas

What are the advantages of microreactor?

- High surface-to-volume ratios, which increases rate of reactions in comparison to macroscopic devices

- High heat and mass transfer rates in microreactors allow reactions to be carried out more aggressively which produces high yields that aren't achievable with conventional reactors.

- Safety. Even if a microreactor were to fail or an exothermic runaway reaction occurred, the small amount of chemicals released could be contained easily

- Environmentally friendly. Less waste is produced as small amounts of chemicals used in microreactor.

- By scaling-out a process, (adding microreactor units instead of scaling-up) failed units can be replaced easily without interfering with other equipment, which is both a maintenance and economic advantage

Microreaction

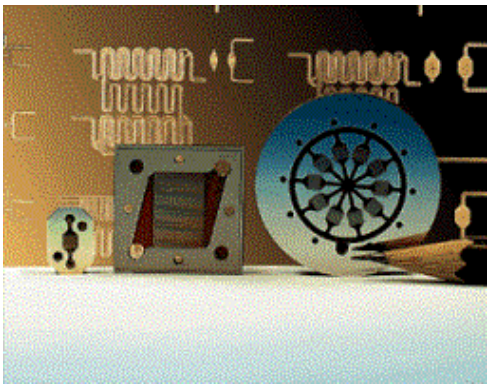

Figure 3: Microreactors. A basic component for microreaction systems is photosensitive glass (Foturan).

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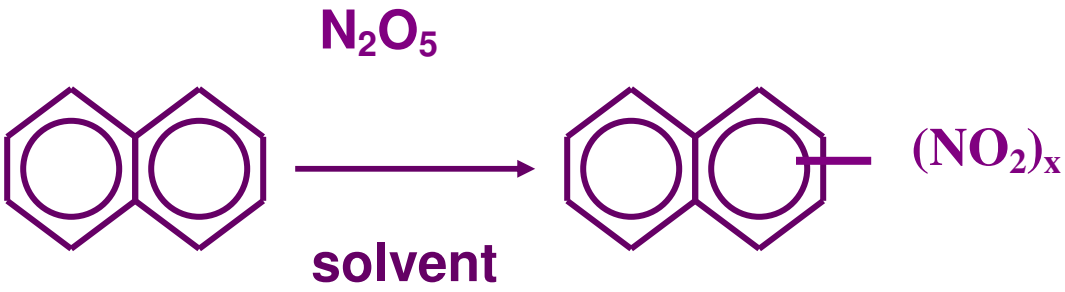
Some common reactions that have been carried out in microreactors include: methanol oxidation, fluorination of toluene, ammonia oxidation, nitration of benzene, ethane epoxidation, and dehydration of methanol to form formaldehyde. At the Prague Institute of Chemical Technology, the chemical engineering department is studying the possibility of enzymatic separations of DNA fragments in microscale chemical devises. Students at the University of Washington are researching explosive mixtures of H2/O2 for catalytic combustion in cross current microreactors. There are many more types of reactions to be explored now that microreactors allow such elevated conditions, not permissible in conventional reactors.

Example for reactions in mikroglas reactor

Direct nitration of aromates (e.g. naphthalene) with dinitrogen pentoxide



Properties of the reaction

- Liquidphase reaction
- Exothermal: DRH0 ca. -500 kJ/mol (explosive !)
- Fast (reaction time ‹ 10 s sufficient)
- Reaction temperature at -50°C (expensive cooling necessary !)
- Complex reaction system (side reactions)

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Result

Conventional technology	**Microreaction technology**
Reaction tends to explosive	Reaction is controlable
Temperature : -50°C	Temperature: +30°C
Mixture of mono-, di-, tri-nitro-product	Only mono-nitro-product

What are micromixers?

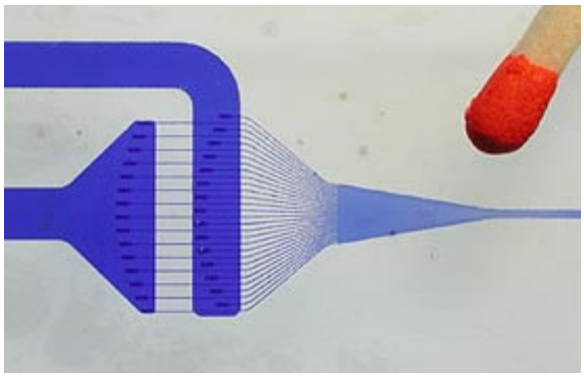

Figure 4 : Interdigital micromixers

Interdigital micromixers made of glass with different outlet geometries were developed in a collaboration between IMM and mgt mikroglas technik AG, Mainz. The figure shows a triangular-shaped interdigital mixer. The mixers made of glass allow the visual investigation of the mixing process and the generation of emulsions.

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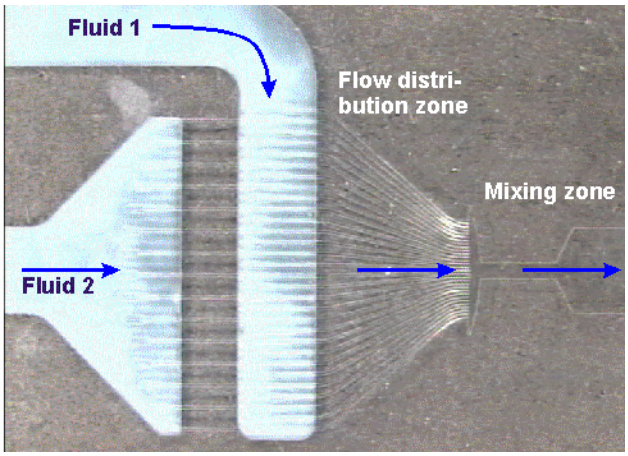

Video sequence:
Visualization of
Dynamic Flow Conditions in a
Slit-Shaped Interdigital Mixer

Example for reactions in mikroglas mixer

- Synthesis of Benzaldehyd from Sulfuric acid and Benzal Chloride

Properties of the reaction

- liquidphase reaction: 2 phases, highly viscous, difficult to mix
- exothermic reaction: cooling to 0°C necessary

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Result

Conventional technology	**Microreaction technology**
Difficult mixing	Fast diffusion controlled mixing
Temperature : 0 °C	Temperature : +70 °C
Yield : 65%	Yield : 69%
Reaction time : several hours	Reaction time : 6 min

What are heat exchangers?

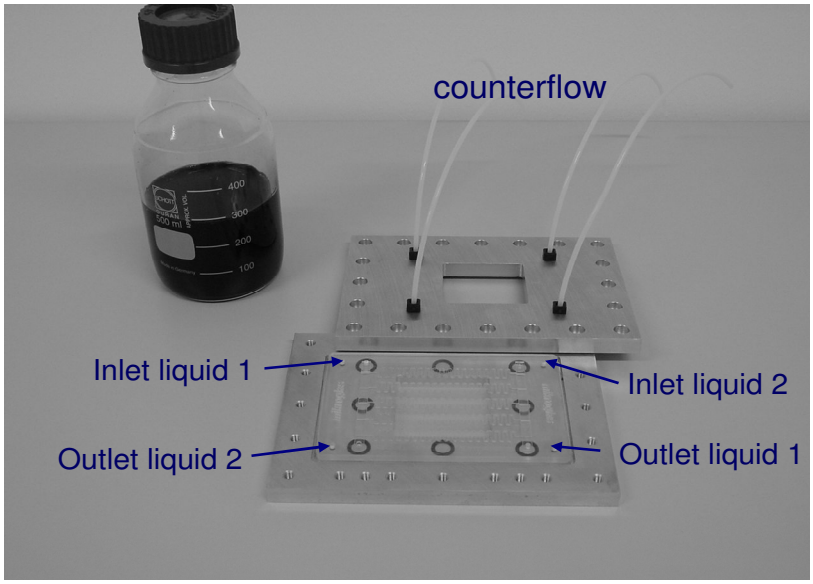

Figure 5: Mikroglas heat exchanger

5 channels run parallel :

- channel width: 1.0 mm
- channel depth: 0.5 mm
- channel length: 280 mm
- wall thickness: 0.2 mm

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To provide the heating and cooling for reactions, heat exchangers are integrated into microreactors. Walls of microchannels have high heat transfer coefficients, usually one magnitude greater than conventional heat exchangers. A common type of heat exchanger integrated into a microreactor is one with stacked sheets of machined channels where hot and cold pass through alternating layers.

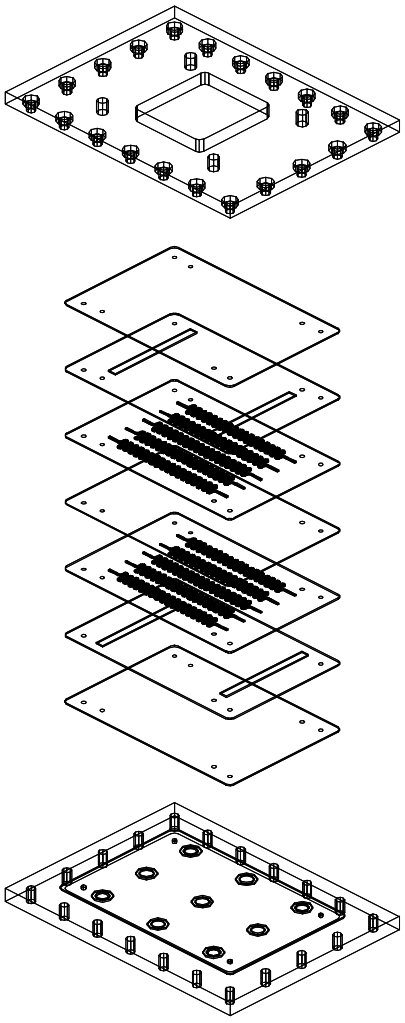

Figure 6 : Sketch- mikroglas heat exchanger

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What is microreaction system?

Figure 7 : microreaction system

Sstem solution which includes:

- ***mikroglas*** - reaction module
- 2 rotary pumps to pump the reagents
  (0.1 ml/min. up to appr. 20 ml/min.) 1 gear pump to pump the tempered media
  (50 ml/min. up to appr. 200 ml/min.)
- Valves
- sensors (temperature and pressure)
- operating unit
- process control by SIEMENS

Microreaction system :

- Mix different media within an adjustable range of volume flows
- Control the reaction temperature by heat exchanger
- Record in-and outlet temperature of the fluid as well as the drop in pressure.

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Mikroglas- Microreaction system

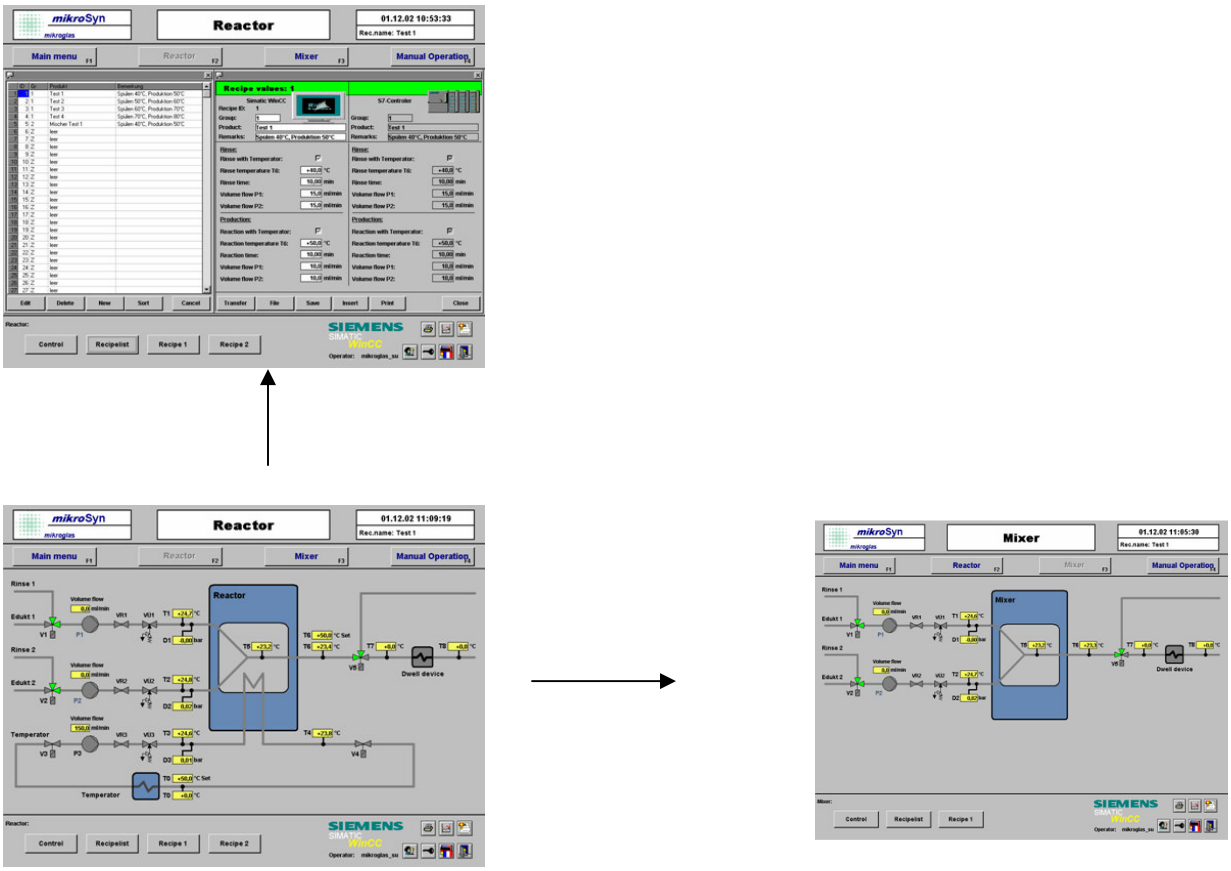

Figure 8: Mikroglas – microreaction system develop together with SIEMENS (SIMATIC S7)

The process control enables the user to monitor different functions of the microreaction system. It is also possible to document the trial parameters precisely.