Microtechnology – New Paradigm For Process industries

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Microproducts Breakthrough Institute

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Microtechnology

The study, development, and application of devices whose operation is based on the scale of 1-100 microns.

(A human hair is approximately 100 microns thick.)

Image source: http://www.flickr.com/photos/thestarshine/69591402/
Nature’s Microtechnology

Nature has selected the micro scale for the realization of many biological processes.

Leaf  Alveoli  Kidney
What is Microtechnology Good For?

- Production of information
  - lab-on-chip
- Production of services
  - pacemaker
  - kidney dialyser
- Production of energy and bulk material
  - chemicals
  - fuels
  - nanoparticles
Micro/Nano Technologies Under Development
In Dr. Jovanovic Laboratory

- Microreactors for Biodiesel Production.
- Microreactors for Production H$_2$O$_2$.
- Microreactor for Desulphurization of Fuels.
- Microseparators for Liquid-Liquid Extraction.
- Microreactors for Production of Veins and Arteries.
- Micro Haemo Dialyser.
- Microreactor for Destruction of Toxic Waste.
- Microseparators for Desalination of Water
- Microreactors for Steam Reforming (atm, 1100°F)
Fundamental Advantages of Microtechnology

• Intensification of Heat and Mass Transport
  - Small scale - Short time of mass and heat transport ($t = l^2/D$)

• Reduced Size
  - 10-100 times reduction in hardware volume over conventional technology;
  - 5-50 times reduction in hardware mass;
  - Shifts size-energy trade-offs toward higher efficiency;
  - Able to integrate heat exchanges with reactors and separators
    simplifying processes.

• Large surface to volume ratio ($10^5-10^8 \text{ m}^2/\text{m}^3$)

• Changes chemical product distribution
Fundamental Advantages of Microtechnology

• **Low Pressure Drop**
  Reduces power for pumps, fans, and blowers;

• **Gravity independence**
  Gravity effect diminish to surface and hydrodynamics forces as size of channels decreases;

• **High Degree of Reaction Control**
  Minimizing unwanted environmental and side reactions;
  Minimize unwanted reversible reactions;
  Enables processing of very energetic reactants;
  Intensification of chemical kinetics (*the last frontier in mass transport*)

• **Extremely High Quench Rates**
  Small reactant volumes mean less mass or energy required to quench;
  Extremely rapid heat transport enables fast thermal discharge.
Advantages of Microtechnology - Parallel Architecture

• Fast screening of materials, catalyst and processes
  
  **Flexibility in capacity and in design**
  - Provides for deployment at wide range of scales;
  - Facilitates gradual expansion of capacity as scale of operations grows by adding more modules;

• Operating robustness and controllability
  - Enhances reliability, allowing problems to be isolated and repaired.

• Mass Production of Microscale Components
  - Microlamination process enables mass production;
  - Bonded stacks can contain multiple processes;
  - Multiple processes in a single device reduces field assembly and testing.
Commercial Advantages of Microtechnology

- Lower capital investment;
- Lower operating cost;
- Faster transfer of research to commercial production;
- Earlier start of production at lower cost
  - Reduces life-cycle costs through early testing at implementation scale;
- Easier scale up (numbering -up) to production capacity;
- Distributed technology implementation (distributed production);
- Integration of micro-technologies with other systems;
- Lower cost of transportation of material and energy;
- Replacing batch with continuous processes.
Safety and Security Advantages

• Small channel inhibits flame/explosion front propagation;
• Small volumes translate to low stored energy;
• Smaller volume less hazardous materials in the process.
Sweet Spot of Microtechnology

- Large surface to volume ratio
- Flexibility in capacity and design
- Distributed production
- Integration with other systems
Micro-Scale Reactors

First MECS micro-reactor, OSU 1999
Catalyst and Catalyst Deposition

Catalyst and Catalyst Deposition
Catalyst and Catalyst Deposition
FeAl 200 µm thick sheet, operating temperature 1100 °C
GoNano Technologies Inc.
121 W Sweet Ave, Suite 115, Moscow ID 83843
Microreactors

- Plate
- Gasket
- Quartz window
- Flow separator
- Teflon spacer
Microreactors
Experimental Setup – Biodiesel Production

Stock solution of methanol with dissolved NaOH in 10 ml syringe

Syringe Pump

Soybean Oil in 60 ml syringe

Microreactor

Biodiesel Phase

Glycerol Phase

ON/OFF
Scale-Up = Numbering-Up
Various Views - Biodiesel Microreactor

Biodiesel
Heating Fluid
Methanol
Crude Oil
Glycerol

Single Stage Biodiesel Microreactor

- Oil Inlet Manifold
- Reaction Channels
- Oil/Glycerol Phase Separation Chamber
- Methanol Inlets
- Glycerol Outlet
- Oil/Biodiesel Phase Outlet to Second Stage
Two Stage Biodiesel Microreactor

First Stage
- Oil Inlet
- Methanol Inlet
- Heating Fluid Inlet/Outlet
- Product Glycerol Stream

Second Stage
- Methanol Inlet
- Product Biodiesel Outlet
- Heating Fluid Inlet/Outlet
- Product Glycerol Stream

Materials:
- Biodiesel
- Heating Fluid
- Methanol
- Crude Oil
- Glycerol
Exploded View - Biodiesel Microreactor

- Second Stage
  - Methanol Inlet/Manifold
  - Oil Phase Outlet
  - Product Biodiesel Outlet
  - Reaction Channels
- First Stage
  - Separation Chamber
  - Oil Phase Outlet
  - Product Glycerol Outlet
  - Methanol Inlet/Manifold
  - Reaction Channels

- Heating Fluid
- Methanol
- Crude Oil
- Glycerol
- Biodiesel

Micromixer Plates

Mixing Channels

Alcohol Feed Channel

Sintered SS Plate
Microreactor Design

Reactor Channels
Separator Plate
Reactor Channels
The Integrated 3-Stage Biodiesel Plant

- **Stage 1**
- **Stage 2**
- **Stage 3**

**Size**
20” X 16” X 8”

**Capacity**
12ml/min = ~4.5 gal/day
1500 gal/year
Oxidative desulfurization of fuels

Thiophene

\[
\text{S}\quad [\text{OH}^+] \quad \text{S} \quad [\text{OH}^+] \quad \text{S}
\]

Non-polar \quad Polar \quad Polar

Dibenzothiophene

\[
\text{S} \quad [\text{OH}^+] \quad \text{S} \quad [\text{OH}^+] \quad \text{S}
\]

Non-polar \quad Polar \quad Polar
The desulfurization reaction kinetics is approximated with a pseudo 1st order rate model. The pseudo 1st order approximation is associated with the overall degradation reaction of thiophene which consist of the following steps:

\[ T \xrightarrow{h\nu, k_1} T^* \]  
**Activation of thiophene**

\[ H_2O_2 \xrightarrow{h\nu, k_2} H_2O_2^* \]  
**Activation of oxidant**

\[ T^* \xrightarrow{k_{-1}} T \]  
**Deactivation of thiophene**

\[ T^* \xrightarrow{k_3} \text{product} \]  
**Activated thiophene conversion**

\[ T + H_2O_2^* \xrightarrow{k_4} \text{products} \]  
**Thiophene conversion with activated oxidant**
Desulfurization of Fuels Two-phase microreactor

- Two reactants enter micro-channel separately with flow rates $Q_1$ and $Q_2$;
- Two phases have different properties ($D, \gamma, \mu, \eta$)

$A + B \Rightarrow R \quad -r_A = kC_A C_B$

Second order chemical reaction;

- $Q_1$
- $Q_2$

Phase 1 - reactant A

Phase 2 - reactant B

UV-light source

UV transparent window

Interface

$A + B \Rightarrow R \quad -r_A = kC_A C_B$

Second order chemical reaction;
Experimental Set-up

Hexane phase
(300 ppm Thiophene/Dibenzothiophene)

Syringe Pump

30% H₂O₂

UV Light

Microreactor

Hexane Phase

Water Phase
Experimental Setup for Interface Reactions
Desulphurization of Fuels
Experimental results

Desulphurization of Fuels

\[ C_0 = 300 \text{ ppm} \]
\[ I = 5.0 \times 10^{-3} \text{ m} \]
\[ D = 3.88 \times 10^{-9} \text{ m}^2\text{s}^{-1} \]
\[ \text{Temp} = 25^\circ\text{C} \]

\[ x = 98.20\% \]
\[ C = 5.40 \text{ ppm} \]

Residence Time (min.)
Experimental Results and Model Simulation

Thiophene concentration at spacer thickness = 50 µm

- $C_0 = 300$ ppm
- $I = 5.0 \times 10^{-3}$ m
- $D = 3.88 \times 10^{-9}$ m$^2$s$^{-1}$
- Temp = 25°C

$x = 98.20\%$

$C = 5.40$ ppm

Residence Time (min.)
Published data by other researchers

- Batch reactor - T - 70°C [20]
- Batch reactor - DBT - 30% H₂O₂ - 50°C - b>280nm [36]
- Batch reactor - 4,6-DMDBT - no H₂O₂ - air=1L/min - 50°C [22]
- Batch reactor - DBT - no H₂O₂ - air=0.5L/min - 50°C [23]
- Batch reactor - DBT - no H₂O₂ - air=1L/min - 50°C [22]
- Batch reactor - DBT - 30% H₂O₂ - 50°C [23]
Comparison with other researchers

OSU Data

- Batch reactor - T - 70oC [20]
- Batch reactor - DBT - 30% H2O2 - 50oC - L 280nm [36]
- Batch reactor - 4,6-DMDBT - no H2O2 - air=1L/min - 50oC [22]
- Microreactor - T - 100 mm
- Microreactor - T - 50 mm
- Batch reactor - DBT - no H2O2 - air=0.5L/min - 50oC [23]
- Batch reactor - DBT - no H2O2 - air=1L/min - 50oC [22]
- Batch reactor - DBT - 30% H2O2 - 50oC [23]
- Microreactor - T - 50 mm
DBT Conversion at 50 µm Homogenous Microreactor

- Temp 22°C
- Temp 40°C

Spacer: 50 µm
Co: 700 ppm
DBT Conversion in 50 µm Microreactor
Microtechnology Based Processes
Steam Reforming of CH$_4$ and Biodiesel

Two 25.0 mm × 7.5 mm × 220 µm microchannels separated by 200 µm catalyst support plate with a catalytic surface of 165 mm$^2$

T=1000°C
Microtechnology Based Processes
Steam Reforming of Hydrocarbons
Microtechnology Based Processes
Steam Reforming of Hydrocarbons

Curtsey of Dr. Al-Khaldi
Microtechnology Based Hemodialyzer
Microtechnology Based Hemodialyzer
Microtechnology Based Desalination Capacitive Deionization Cell (CDT)
We expect the following advantages of Capacitive Desalination over best commercially available technology (RO Desalination)

- The concept can approach the theoretical limit on minimum energy consumption; initially, we are projecting energy consumption of 1.5 – 4.0 [kWh/m³] fresh water produced;
- Flexible capacity – from small to very large units (numbering-up v.s. scale-up)
- Possibility of use of renewable energy at the point of use (solar/wind)
- Lower capital investment;
- Lower operating cost;
Typical Development Steps

• Build and demonstrate a nominal size technology unit.

• Develop inexpensive large scale manufacturing technology for microscale devices.

• Educate new generation of PhDs capable of introducing Microtechnologies worldwide.

• Launch new small business ventures and create jobs.
Ensuring a sustainable future requires well-educated students who are not afraid of new technological world.

- Kasidid Asumingpong, M.Sc.
- James Parker, Ph.D
- Joy Das, M.Sc.
- Daniel Haller, Ph.D.
- Eric Anderson, Ph.D.
- Brian Reed, Ph.D.
- Eileen Hebert, M.Sc.
Thank you for your attention!