Continuous Chemical Synthesis within the ResonantAcoustic® Mixing Platform

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Technical InterChange 2025

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Objectives

- Understand the challenges in reactor design
- Describe behavior of liquid and gas mixing in a ResonantAcoustic® Continuous Flow Device
- Demonstrate benefits of using ResonantAcoustic® Continuous Flow Device for Chemical Reactions
  - Improved Mass Transfer (Decreased Mixing Time)
  - Increased Reaction Rate for Mixing Limited Reactions
  - Improved Heat Transfer
  - Able to process multiphase processes
  - Scalable Process

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What Makes a Good Reactor Design?

- High Conversion, High Yield
- Fast, Complete Mixing of Reactants
- Temperature Control
- Consistent Results
- Flexible to range of operations
- All physical phenomena are well understood
- Physical phenomena are scalable

Example Rate Expression

$$r_P = k_1 C_A C_B$$

Optimized Temperature (k_1)
Optimized Mixing ($C_A C_B$)

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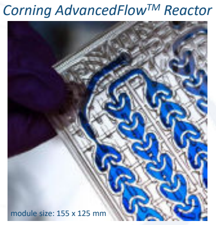
Legacy Batch vs. Advanced Flow Reactors

Chemglass 10L Reactor

Corning AdvancedFlow™ Reactor



Conventional Batch Reactors:
+ Inexpensive and Flexible
+ Multiphase Processes
- Variability on product quality
- Mixing and Heat-Exchange Limitations

Microfluidic Reactors:
+ Improved Mixing and Temp Control
+ Control and Tunability of Product
+ Decreased Waste
- Complexity and Cost
- Difficulty Processing Solids

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ResonantAcoustic® Mixing Phenomenon

Dow 200 @ 1,000 cP
90 *g* of acceleration

11 inches

14 inches

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Basic Principles of Gas-Liquid Mixing

- Faraday Instabilities Provide a Mechanism for Gas Bubble Encapsulation and Liquid Droplet Formation
- Once Droplets and/or Bubbles Are Created, Acoustic Waves Continue to Create Differential Motion Between the Two Phases
- Disparate Densities of Liquids and Gases Result in Excellent Mixing from RAM Motion

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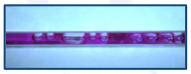
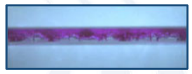
Continuous Gas-Liquid Mixing Regimes

Compressive Regime vs. Splitting and Combining Regime



0 *g* Acceleration, 150 mL/min
4 mm Inner Diameter
Re = 780 → laminar flow

40 *g* Acceleration, 150 mL/min



100 *g* Acceleration, 150 mL/min

Gas Headspace is Required for RAM Enhanced Mixing

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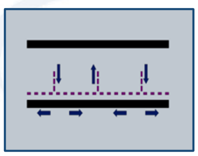
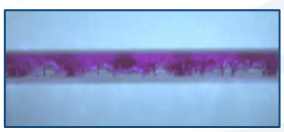
Splitting and Combining Regime

Increases Surface Area and Reduces Boundary Layer Thickness





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Demonstration of Minimal Back-Mixing

1% Xanthan Gum Solution (200 cP)
95g, 50 mL/min, 60 frames per second

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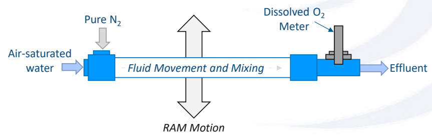
Mass Transfer Coefficient Measurement

- Dissolved Oxygen Experiments:
  - Air-saturated water fed to 4mm reactor
  - Pure N_2 also fed at reactor inlet
  - Dissolved-oxygen meter measures transfer of O_2 into the gas phase

$$k_L a = \frac{1}{t} \ln \frac{c_{in} - c_{equ}}{c_{out} - c_{equ}}$$



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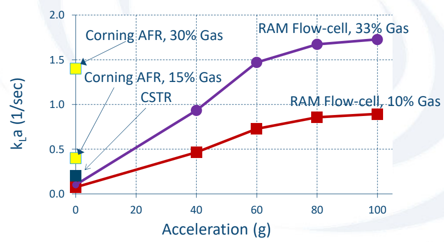
Mass Transfer Coefficient Measurement

- Dissolved Oxygen Experiments:
  - Corning AFR is 7-fold improvement in Mass Transfer compared to legacy CSTR
  - Continuous ResonantAcoustic® Mixing is a further 35% improvement in Mass Transfer with an 88% lower residence time



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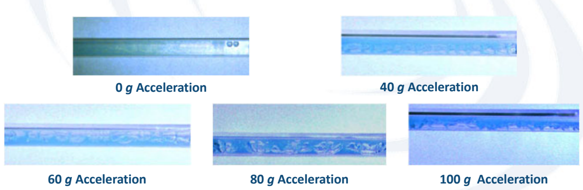
Precipitation Reaction Demonstration

- Advanced microreactors easily clog in the presence of solids
- Continuous ResonantAcoustic® process can handle solids
- Example: Formation of Basic Copper Carbonate solids

$$2\,Cu^{2+}(aq) + 2CO_3^{2-}(aq) + H_2O \rightarrow Cu_2(OH)_2CO_3(s) + CO_2(g)$$



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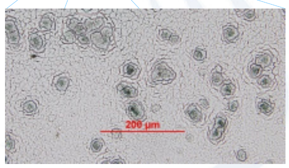
Precipitation Reaction Demonstration

Sample collected from microreactor 80-100 *g* acceleration

After Centrifuging

Solids Recovered from Centrifuge Vial



Microscope Images Reveal Basic Copper Carbonate Particles

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Model Reaction Study: Mixing Time Measurement

- Parallel Competitive Reactions to quantify mixing time

$$H_2BO_3^- + H^+ \overset{fast}{\longleftrightarrow} H_3BO_3$$

$$IO_3^- + 5I^- + 6H^+ \overset{slower}{\longleftrightarrow} 3I_2 + 3H_2O$$

$$I_2 + I^- \leftrightarrow I_3^-$$

- Fast reaction consumes acid when perfectly mixed
- Any degree of poor mixing, side reaction will form I_2 formation
- UV/VIS measurement of $[I_3^-]$ in product, used to calculate mix quality and mixing time

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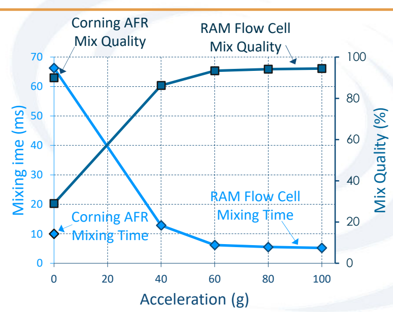
Model Reaction Study: Mixing Time Measurement



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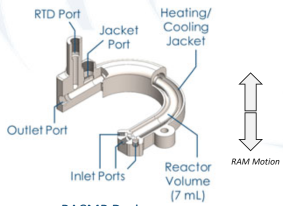
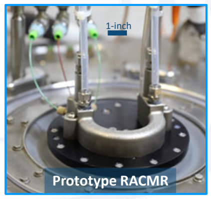
ResonantAcoustic® Continuous Microreactor

- RACMR Prototype:
  - Incorporation of up to three reactants
  - Immediate and complete mixing of reactants
  - RAM results in plug flow fluid profile
  - Jacketed for cooling or heating of reaction mixture (4,400 W/m^2-K)



RACMR Design



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Summary

- Rapid and complete incorporation of feeds (mixing time: 6 ms)
- Highly effective heat transfer (4,400 W/m^2-K)
- Mass/Heat Transfer independent of flow velocity
- Ability to handle slurries, viscous flow, multi-phase processes, etc.
- Scalable process from bench-scale to industrial-scale reactors

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