

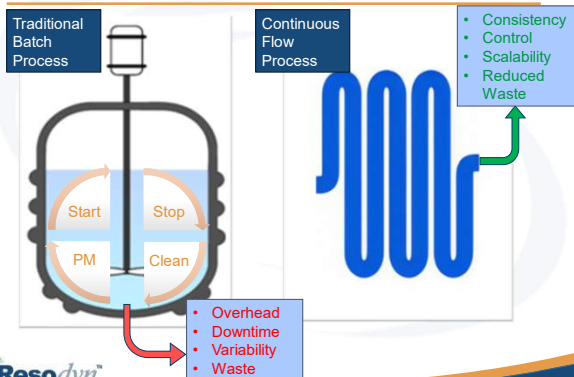
Continuous Chemical Synthesis Using Resonant Acoustic® Reactors

Seth Pattee, M.S.
New Product and Process Development Chemist
Resodyn Corporation



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Why Choose Continuous Chemical Manufacturing?



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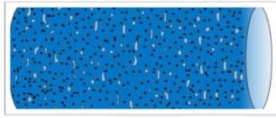
What Makes a Great Continuous Reactor Platform?



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Challenges in Continuous Flow Processing

- Elevated-viscosity materials
- Multiphase systems
- Slurry feeds
- Gas-evolving reactions
- Precipitation reactions



How do we navigate these challenges?



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ResonantAcoustic® Mixing Phenomenon



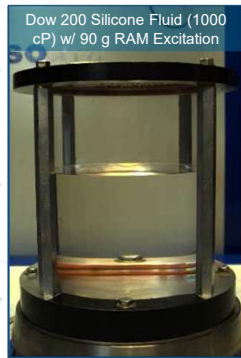
Surface
Instability
Formation



Differential
Motion



Bulk Fluid
Circulation



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Continuous Gas-Liquid Mixing Regimes

Compressive

Splitting & Combining



0 g Acceleration
(Laminar Flow)

40 g Acceleration
-Gas phase begins to subdivide
-Increased gas/liquid interaction

100 g Acceleration
-Extensive phase subdivision
-Dynamic gas-liquid interaction

Conditions: 150 mL/min flow, 4 mm ID reactor, $Re = 780$

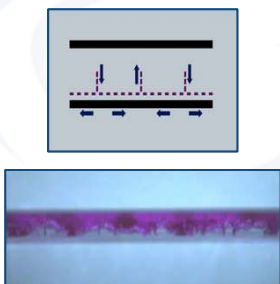
A gas phase is required to achieve RAM-enhanced mixing within the continuous reactor.



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Splitting and Combining Regime

Increases Surface Area and Reduces Boundary Layer Thickness



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Rapid Mixing with Limited Back-Mixing

Conditions: 1 wt% Xanthan Gum (~200 cP), 95 g Acceleration, 50 mL/min flow



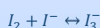
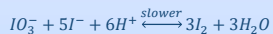
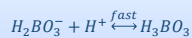
- Dye is rapidly incorporated but remains localized within the reactor indicating limited back-mixing.

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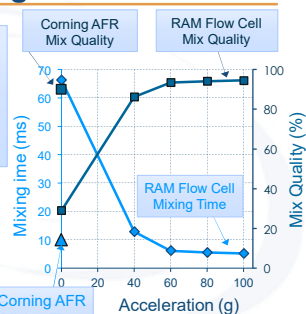
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Quantifying Mixing Performance

Villermoux-Dushman Reaction



- Parallel competitive reactions used to assess mixing
- Poor mixing produces the diiodine (I_2) species through side reaction
- UV/Vis measurement of I_2/I_3^- used to estimate mixing quality and time



RAM Flow Cell exceeds Corning Advanced Flow Reactor performance!

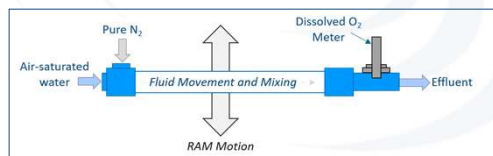
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Quantifying Gas-Liquid Mass Transfer

- Dissolved Oxygen Experiments
 - Air-sat. water fed to 4mm RAM reactor
 - N₂ gas introduced at reactor inlet
 - Dissolved O₂ measured in effluent
 - Volumetric mass flow coeff. (k_La) determined from O₂ transfer

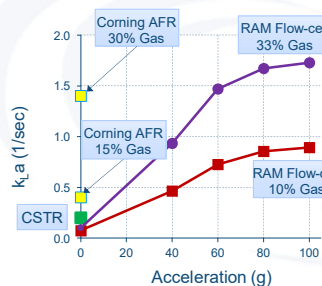
$$k_L a = \frac{1}{t} \ln \frac{c_{in} - c_{equ}}{c_{out} - c_{equ}}$$



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Quantifying Gas-Liquid Mass Transfer



Conclusions

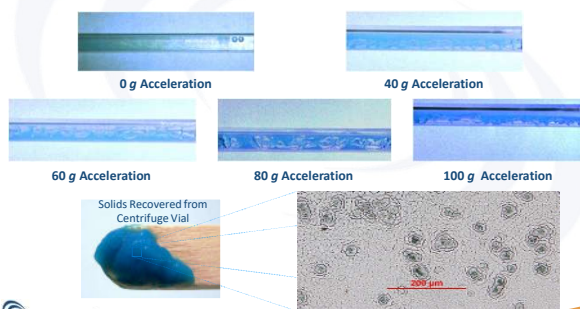
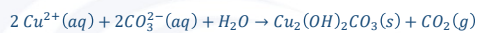
- Continuous RAM saw 35% improvement in mass transfer over AFR with 88% lower residence time

Mass transfer performance increased with acoustic mixing and exceeded both the Corning AFR and a conventional CSTR under conditions evaluated.

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Continuous Processing of Solids-Forming Reactions



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Case Study 1: Continuous Energetic Precursor Synthesis



Project Goal: Demonstrate continuous synthesis of key energetic precursor

Design Strategy:

- LabRAM IIR: 7mL RACMR, 2°C
- PharmaRAM II: 70mL Coil Reactor, 40°C

Desired Reactions

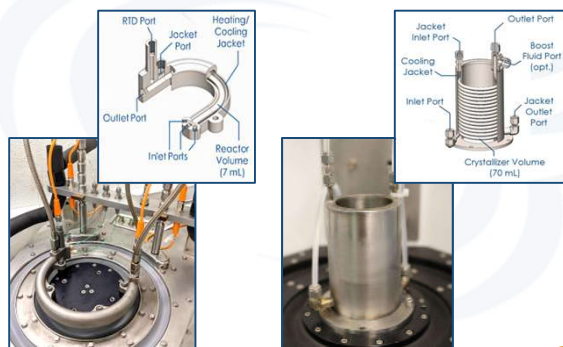


Undesired Reactions



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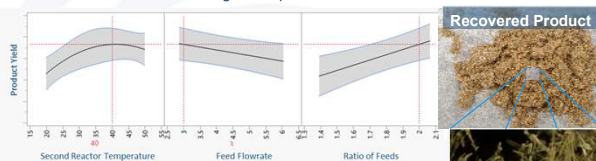
Case Study 1: Continuous Energetic Precursor Synthesis



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Case Study 1: Continuous Energetic Precursor Synthesis

Design of Experiment Results



DOE Summary:

- **Temperature:** Yield ↑ to optimum near 40°C
- **Flow Rate:** Yield ↓ as flow rate ↑
- **Feed Ratio:** Yield ↑ as feed ratio ↑

Process Performance:

- **99.5% Product Purity**
 - Literature: 60-78%
- **90% Product Yield**
 - Literature: 40-65%
- >700 min Continuous Operation
- >1 kg Product Delivered to NSWC-IH



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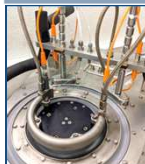
Case Study 2: High-Temperature/Pressure Synthesis

Project Goal: Pilot continuous synthesis of key energetic precursor

Project Challenges:

- Modified Strecker synthesis
 - Slower, heat-absorbing chemistry
 - Competitive side-reactions for a polymer byproduct
 - Limited academic literature
- High-Temperature RACMR
 - 18mL Volume
 - 200°C Temp
 - 40 bar pressure
 - Integrated temp measurements
 - 24-hour Continuous Operation

LLNL RACMR



Lawrence Livermore
National Laboratory

>90% Yield
>90% Purity



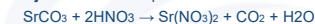
Redesigned RACMR enabled continuous synthesis at elevated temperature & pressure while maintaining product quality and avoiding problematic byproducts

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Case Study 3: Continuous Processing with Slurry Feed and Gas Evolution

Project Goal: Develop and demonstrate continuous system for $\text{Sr}(\text{NO}_3)_2$ synthesis



Project Challenges:

- Flexible platform for multiple defense-critical materials
- Slurry-reactant feed
- Carbon dioxide generated during reaction
- Continuous solids handling throughout

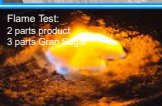
Process Results:

- 100% Conversion
- 99.6% Purity
- 7 g/min Continuous Production
- Demonstrated membrane-based gas removal



RACMR Membrane
Reactor

$\text{Sr}(\text{NO}_3)_2$ Product



Flame Test:
2 parts product
3 parts Gas

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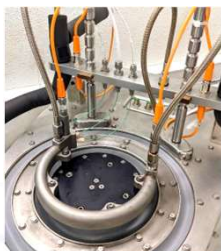
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Summary of Capabilities

- Gas-Liquid Mass Transfer
- Limited Back-Mixing
- Solids Precipitation & Slurries
- Multi-Step Synthesis
- High Pressure/Temp Synthesis
- Multiphase Synthesis (Gas + Liquid + Solid)



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Where does ResonantAcoustic® Processing Fit?



- Plug-Flow/Tubular Reactor (PFR)**
- High conversion in liquid-liquid regimes/scalable
 - Susceptible to fouling and plugging



- Microreactors**
- Exceptional heat and mass transfer
 - High pressure drop & solids sensitivity



- Continuous Stirred-Tank Reactor (CSTR)**
- Long residence times & solids-tolerant
 - Low conversion per unit volume



- ResonantAcoustic® Chemical Reactor**
- Multiphase functionality & high-mixing intensity
 - Process-specific reactor optimization may be required



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Key Takeaways

- ResonantAcoustic® Processing:
 - Enhances fundamental reactor performance
 - Demonstrated across difficult reaction regimes
 - Expands the continuous manufacturing toolbox



ResonantAcoustic® Technology Enables Continuous Chemical Processing in Challenging Environments



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**Thank you for your time
and attention.**



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