High Shear Mixing Research Program
Introduction & Overview

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BHR Group DOMINO Project Meetings
23 September 2015
High Shear Mixing Research Program Participants

- Bristol-Myers Squibb
- Chevron ETC
- Dow Chemical/Rohm & Haas
- Dow Corning
- DuPont Engineering
- Eli Lilly & Co.
- FL Schmidt
- Koch Materials Co.
- LG Chemicals
- Merck & Co.
- Pfizer
- Procter & Gamble
- Syngenta/Zeneca
- ANSYS - FLUENT
- PSE (Process Systems Enterprise Ltd.)
- Charles Ross & Son
- Chemineer/Greerco
- IKA Works
- Lightnin
- Silverson Machines
- Lenterra
- BHR Group FMP
High Shear Mixer Studies
Mostly Turbulent Flow

• Single phase (water) flow field characterization in in-line (continuous) and batch rotor-stator mixers
  – Computational Fluid Dynamics (CFD) simulations
  – Measurements via Laser Doppler Anemometry (LDA)
  – Measurements via Particle Image Velocimetry (PIV)

• DSD for dilute liquid-liquid dispersions in batch rotor-stator mixers
  – Drop size data as a function of geometry, operating conditions and relevant physicochemical properties
  – Mechanistic models to guide data interpretation
  – Effect of surfactants on drop deformation and breakup

• Single pass DSD for in-line rotor-stator mixers
  – Dilute liquid-liquid dispersions
  – Single and multistage mixers
High Shear Mixer Studies
Laminar and Turbulent Flow

• Power draw measurements
  – Batch and in-line rotor stator mixers
  – Aid in interpretation of DSD data

• DSD for more concentrated dispersions
  – Batch rotor-stator mixer
  – Water into oil dispersions

• Crystal size reduction via wet milling
  – In-line rotor stator mixer
  – Operated in the recirculation loop of a stirred vessel
  – Multi-zonal population balance
Complementary Studies

Turbulent Flow

• Similar studies for stirred tanks and static mixers

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Complementary Studies

Laminar Flow

• Drop breakup in simple shear flow (SSF)
  – Couette device

• Break up of a surfactant laden laminar jet
  – Water into air
  – Oil into water

• Breakage of encapsulated drops (Chevron ETC)
  – Parallel plate flow channel
Independent Industrial Grants

• Pfizer Inc. Strategic Alliance
  ➢ Mechanisms for and Scale-up of High Shear Wet Milling of Pharmaceutical Ingredients
  ➢ Computational Fluid Dynamics - bench, pilot & plant scales
  ➢ Wet milling of crystals on bench scale – preliminary experiments

• Chevron Energy Technology Company (ETC)
  ➢ High Shear Effects on Water in Oil Emulsions
  ➢ Effect of physicochemical properties, water phase fraction, solid surface wetability and agitation rate on the size of water drops
  ➢ Breakage of Encapsulated Drops

• Lenterra - Wall Shear Stress Sensor
  ➢ NSF SBIR Phase 1 & 2 Demonstration/Development Grants
  ➢ Demonstrated sensor in parallel plate device
    ▸ Compared measured stress to CFD predictions
  ➢ Upgraded IKA prototype mixer facility to serve as main test bed
In-Line Rotor-Stator Mixers

• IKA prototype with one row of rotor and one row of stator teeth

• Greerco Tandem Pipeline Mixer

• Silverson L4R with in-line attachment

• IKA Labor Pilot 2000/4 Benchtop, 3 stage in-line modular unit
Stator slot: 6 x 10 x 12 mm

Rotor slot: 10 x 10 x 10 mm

Gap Width: 0.5 mm

Outer diameter of stator: 154 mm

12 rotor & 14 stator teeth

IKA Prototype In-Line Rotor-Stator Mixer
IKA Prototype In-Line Rotor-Stator Mixer
IKA Labor Pilot 2000/4 Benchtop, 3 Stage In-Line Modular Dispersion Unit

- Inlet
- Outlet
- 1 to 3 stages
- 1.5 gpm max

Generator Accessories:
- General
- Coarse
- Medium
- Fine
- Ultrafine
Greerco Tandem Pipeline Mixer
2 inch unit installed in ChE-1235 Flow Loop
RANS CFD Simulation of 4 inch unit currently underway

Stage 1
Stage 2

Rotor (stage 1)
Stator
Batch Rotor-Stator Mixers

• Silverson L4R mixer with integrated glass vessel
  – Different unit than one with in-line attachment

• Ross ME 100 LC with integrated stainless steel vessel

• Greerco Model XLR Homogenizer
Silverson L4R Batch Rotor Stator Mixer

Silverson Model L4R
High Shear Mixer
2.0 liter glass vessel
4 Blade rotor, diameter = 2.8 cm
R-S gap width = 0.203 mm
Ross ME 100 LC Batch Rotor Stator Mixer

Ross Model ME 100 LC
High shear mixer
2.5 liter vessel
4 Blade rotor, diameter = 3.4 cm
R-S gap width = 0.5 mm
Greerco XLR Batch Homomixer

Axial Flow

Rotor diameter = 4.3 cm
R-S gap width = 0.5 mm
11.5 liter open glass vessel - variable
Computational Fluid Dynamics

- ANSYS - Fluent, Inc.
  - RANS MRF and sliding mesh simulations
  - Particle Tracking (DPM)
  - Fast Particle Tracking Algorithm (in-house)
    - IKA Prototype & Silverson L4R, 450 LS & 600 LS
      In-line Mixers
    - Silverson L4R Batch Mixer
    - Greerco Tandem Pipeline Mixer

- ANSYS - Fluent, Inc.
  - Large Eddy Simulation of Virtual Impactor
    - Fluent UDF for Lagrangian Dynamic Model

- AcuSim Software, Inc.
  - Large Eddy Simulation of IKA Prototype In-line Mixer

- Meshing Solutions:
  - Harpoon (Sharc Ltd) & TGrid wrapper (Fluent)
  - ANSYS Workbench, etc.
In-Line Rotor-Stator Measurement Techniques

• Laser Doppler Anemometry (LDA)
  – Mean velocity field and statistical turbulence parameters
  – Direct comparison to RANS sliding mesh simulations

• Phase Doppler Anemometry (PDA)
  – Particle mean velocities & statistical turbulence parameters
  – Particle size distribution
  – Same platform as LDA

• Particle Image Velocimetry (PIV)
  – Instantaneous (time resolved) ‘full’ velocity fields
  – Direct measurement of energy dissipation rate possible
  – Direct comparison to LES

• High speed video imaging
Batch Rotor-Stator Measurement Techniques for Turbulent Liquid-Liquid Dispersions

- DSD via High Magnification Video Probe (HMVP), Video Microscope (VM) and Dynamic Light Light Scattering (DLS).
  - HMVP: 5+ to 150 µm  In-situ measurement
  - DLS: 1 nm to 1 µm  No sampling & stabilization issues
  - VM: “in between”  Sampling & stabilization issues Surfactants used to stabilize DSD

- High speed video imaging.

- Dynamic and static interfacial tension via pendant drop techniques - allows estimates of interfacial properties. Approximate measurements of contact angle.

Power Draw Measurements

• Silverson, Ross & Greerco Batch Mixers
  – Comprehensive Power number data
  – Detailed analysis to insure proper definition of Reynolds and Power numbers
  – Used to quantify liquid-liquid dispersion performance
  – Data have been compared to CFD predictions
  – Data used to estimate power draw for in-line mixers

• IKA Labor Pilot 2000/4 In-line Multistage Mixer
  – Comprehensive data for water for various mill heads
  – Used to analyze single pass drop size data
Liquid-Liquid Dispersion Fundamentals

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Case for Dilute Systems

• Coalescence and suspension can be ignored
• Single phase fluid dynamics concepts can be used to quantify the forces acting to deform and break drops
  – Except close to their surface, the presence of drops does not affect continuous phase hydrodynamics.
• Above simplifications allow a quantitative analysis of the DSD
• Fundamental knowledge of drop breakup can be applied to higher concentration systems
• Mechanistic theories can be applied to systems stabilized against coalescence
• DSD data relatively easy to acquire
  – Drop size is spatially uniform
  – Drops are readily imaged in-situ
  – Measured distribution is relatively unambiguous
Breakup of Inviscid Drops in Turbulent Flow

Disruptive Force: \( \tau_c \sim \rho_c v'(d)^2 \)
(Turbulent Stress)

Stabilizing Force: \( \tau_s \sim \sigma/d \)
(Interfacial Tension)

Maximum Stable Drop Size:
\[ \tau_c(d_{\text{max}}) = \tau_s(d_{\text{max}}) \]

Since all drops of size \( d > d_{\text{max}} \) will eventually break, the equilibrium DSD will consist of drops of size \( d_{\text{max}} \) and smaller.
Model for Stress on Drop

Assumption: Ultimate drop size is determined by drop interactions with **Inertial Subrange** eddies

\[ \tau_c = 1.5 \beta \rho_c \varepsilon^{2/3} d^{2/3} \quad (\eta << d << \ell) \]

\( \varepsilon = \text{local maximum near mixing head} \quad (\ell \sim D) \)

Kolmogorov microscale: \( \eta = (\nu_c^3/\varepsilon)^{1/4} \)

Estimate \( \eta \) from power draw data: \( \varepsilon = \frac{P}{\rho_c V_{\text{MH}}} \)

\( \varepsilon \) can also be estimated from LDA/PIV data & CFD simulations

\[ \varepsilon \approx \frac{k^{3/2}}{\ell} \quad \varepsilon = \nu \dot{\gamma}^2 \]
Correlation for Equilibrium Mean Drop Size

Inertial Subrange: \((\eta << d << \ell)\)

General: 
\[ d_{\text{max}} \sim \sigma^{3/5} \rho_c^{-3/5} \varepsilon^{-2/5} \]
(Device Independent)

Constant Power No.: 
\( \varepsilon \propto P/m \propto N^3 D^2 \)
(Geometric Similarity)

Weber No.: 
\[ \text{We} = \frac{\rho_c N^2 D^3}{\sigma} \]

\( d_{32} \propto d_{\text{max}} \)
Correlation for Equilibrium Mean Drop Size

\[
\frac{d_{32}}{D} = 0.053 \text{ We}^{-3/5}
\]

\[
d_{32} = 0.64 d_{\text{max}}
\]

Data of Chen and Middleman (1967) for standard Rushton turbines in baffled cylindrical tanks.

Mechanistic theory also confirmed for ‘long’ static mixers, rotor-stator mixers and other devices.
Other Dispersion Devices

Kenics Static Mixer

Silverson Model L4R Rotor-Stator Mixer

\[ \varepsilon \sim V S^3 / D \]

\[ We = \frac{\rho_c V^2 S D}{\sigma} \]

\[ We = \frac{\rho_c N^2 D^3}{\sigma} \]

\[ \varepsilon \sim N^3 D^2 \]
Correlation for Equilibrium Mean Drop Size

Other Devices:

• Kenics Static Mixer
  – Berkman and Calabrese (1986)
  – Weber No. \( N_{\text{we}} = \rho_c V_s^2 D/\sigma \)
  \[ \frac{d_{32}}{D} = 0.49 \text{ We}^{-3/5} \]
  \[ d_{32} = 0.67 \ d_{\text{max}} \]

• Ross & Silverson Rotor-Stator Mixers
  – Slotted stator head
  – Equal power per stator slot
  – Phongikaroon et al. (2001)
  \[ \frac{d_{32}}{D} = 0.038 \text{ We}^{-3/5} \]
  \[ d_{32} = 0.44 \ d_{\text{max}} \]

• Modify available correlations to apply to other geometries via ratio of Power numbers or local energy dissipation rates

Scale-up and correlate on local power per unit mass:

\[ d_{32} \sim d_{\text{max}} \sim \sigma^{3/5} \rho_c^{-3/5} \varepsilon^{-2/5} \]
Relation of $d_{\text{max}}$ to Local Maximum Power

$$d_{\text{max}} \sim \varepsilon^{-2/5}$$

$d_{\text{max}}$, $\mu$m

Local Power, w/kg

Corrected for $\sigma$

- Static Mixers
- Agitated Vessels
- Colloid Mills
- Liquid Whistles
- Valve Homogenizers
- Ultrasonics

$$\varepsilon \approx \frac{P}{\rho_c V_{\text{DispZone}}}$$

$$\varepsilon = \nu \dot{\gamma}^2$$

$$\varepsilon \approx \frac{k^{3/2}}{\ell}$$
Turbulent Flow - Viscous Dispersed Phase

- **Maximum Stable Drop Size:**
  \[
  \tau_c = \tau_s + \tau_d \quad \text{for } d = d_{\text{max}}
  \]

- **Cohesive Forces**
  
  - **Interfacial Tension:**
    \[
    \tau_s \sim \sigma / d
    \]
  
  - **Drop Viscosity:**
    \[
    \tau_d \sim \mu_d \frac{\sqrt{\tau_c / \rho_d}}{d}
    \]

- **Sauter Mean Diameter:**
  \[
  d_{32} \propto d_{\text{max}}
  \]

- **Geometric Similarity:**
  \[
  \varepsilon \propto P/m \propto N^3 D^2 \text{ or } V_s^3 / D
  \]
Correlation for Equilibrium Mean Drop Size

Inertial Subrange: \((\eta \ll d \ll \ell)\)

General:
\[
\frac{\rho_c \varepsilon^{2/3} d_{32/\text{max}}^{5/3}}{\sigma} = C_1 \left[ 1 + C_2 \left( \frac{\rho_c}{\rho_d} \right)^{1/2} \frac{\mu_d \varepsilon^{1/3} d_{32/\text{max}}^{1/3}}{\sigma} \right]
\]

Geometric Similarity:
\[
\frac{d_{32/\text{max}}}{D} = C_3 \text{ We}^{-3/5} \left[ 1 + C_4 \text{ Vi} \left( \frac{d_{32/\text{max}}}{D} \right)^{1/3} \right]^{3/5}
\]

Viscosity Group:
\[
\text{Vi} = \left( \frac{\rho_c}{\rho_d} \right)^{1/2} \mu_d N D/\sigma \quad \text{or} \quad \text{Vi} = \left( \frac{\rho_c}{\rho_d} \right)^{1/2} \mu_d V_s/\sigma
\]

\(\text{Vi} \to 0:\quad d_{32/\text{max}} \sim \varepsilon^{-2/5} \quad \& \quad \sigma^{3/5}
\)

\(\text{Vi} \to \infty:\quad d_{32/\text{max}} \sim \varepsilon^{-1/4} \quad \& \quad \mu_d^{3/4}
\)

Empirical Constants for Sauter Mean Diameter

Rushton Turbine: \(C_3 = 0.054; \quad C_4 = 4.42\) \(\text{Wang \& Calabrese (1986)}\)

Kenics Static Mixer: \(C_3 = 0.49; \quad C_4 = 1.38\) \(\text{Berkman \& Calabrese (1988)}\)

Rotor-Stator Mixer: \(C_3 = 0.018; \quad C_4 = 13.0\) \(\text{Phongikaroon et al. (2001)}\)
Model Summary: Various Breakup Regimes

Inviscid Drops

The expression for the disruptive force depends on the drop size, so other less common mechanistic correlations can be derived:

- $\eta < d < \ell$: Inertial Subrange model
  \[
  d_{32/\text{max}} \sim \frac{\sigma^{3/5}}{\rho_c^{3/5} \varepsilon^{2/5}}
  \]
  \[
  \frac{d_{32/\text{max}}}{D} \sim \text{We}^{-3/5}
  \]

- $d < \eta$: sub-Kolmogorov inertial stress model
  \[
  d_{32/\text{max}} \sim \frac{\sigma^{1/3} \mu_c^{1/3}}{\rho_c^{2/3} \varepsilon^{1/3}}
  \]
  \[
  \frac{d_{32/\text{max}}}{D} \sim (\text{We Re})^{-1/3}
  \]

- $d << \eta$: sub-Kolmogorov viscous stress model
  \[
  d_{32/\text{max}} \sim \frac{\sigma}{(\rho_c \mu_c \varepsilon)^{1/2}}
  \]
  \[
  \frac{d_{32/\text{max}}}{D} \sim \text{We}^{-1} \text{ Re}^{1/2}
  \]

Reynolds Number:

\[
\text{Re} = \rho_c N D^2 / \mu_c \quad \text{or} \quad \text{Re} = \rho_c V_S D / \mu_c
\]
Inertial to sub-Kolmogorov Correlations

Inviscid Drops

Equilibrium mean drop size for organics and oils dispersed in water /glycerol and water dispersed in oils with slotted stator head
Power Number Correlation to Estimate Local Value of $\varepsilon$

Batch, Radial Flow Lab Scale Rotor-Stator Mixers

\[ N_P = \frac{P'}{\rho_c N^3 D^5} \]

\[ \text{Re} = \frac{N D^2}{v_c} \]

Const. $N_p$: $\varepsilon = \frac{P'}{m} \sim N^3 D^2$
Inertial Sub subrange Correlation

Slotted Stator Head

$\eta < d < \ell$

**Ross Mixer**
- Organics in water

**Ross Mixer**
- Chlorobenzene in aqueous glycerol

**Silverson Mixer**
- Chlorobenzene in water

Geometric similarity based on power draw per stator slot

$D_{32}/L = 0.039 N_{We}^{-3/5}$

$R^2 = 0.85$

$D_{32} = 0.44 d_{max}$

$N_{We} = \frac{\rho c N^2 D^3}{\sigma}$
At low speed, $D_{32}$ decreases as the continuous phase viscosity increases.

\[ \eta = \left( \frac{\nu^3}{\varepsilon} \right)^{1/4} \]

\[ \varepsilon \sim N^3 D^2 \]
Sub-Kolmogorov Viscous Stress Correlation

Slotted Stator Head

\[ \frac{D_{32}}{L} = 0.0037 \left( N_{Re}^{1/2} N_{We}^{-1} \right) \]

\[ R^2 = 0.79 \]

\[ D_{32} = 0.44 \, d_{max} \]

- Organics in water
  Ross mixer
- Chlorobenzene in aqueous glycerol
  Ross mixer

\[ N_{Re} = \frac{\rho_c N D^2}{\mu_c} \]
Water into Oil Dispersions - Silverson Mixer

Data in turbulent regime

\[
\frac{d_{32}}{D} = 0.115 (\text{We Re})^{-1/3}
\]

sub-Kolmogorov inertial stress model

\[
\eta = (v^3/\varepsilon)^{1/4}
\]

Water dispersed in Crystal Oil FG

\[ \phi = 0.001 \]

Slotted Stator Head

Range of \( \eta \), when high-shear region = mill head

Range of \( \eta \), when high-shear region = disk with twice the diameter of mill head

Range of \( d_{\text{max}} \), calculated using \( d_{32} = 0.49 \cdot d_{\text{max}} \)

\[
\begin{array}{|c|c|c|}
\hline
\text{Range of } \eta \text{, when high-shear region = mill head} & \text{Range of } \eta \text{, when high-shear region = disk with twice the diameter of mill head} & \text{Range of } d_{\text{max}}, \text{ calculated using } d_{32} = 0.49 \cdot d_{\text{max}} \\
\hline
[40, 140] \mu m & [57, 200] \mu m & [10.6, 48.6] \mu m \\
\hline
\end{array}
\]

Fig. 13 – Comparison of the turbulent flow drop size data with the sub-Kolmogorov inertial stress model. This plot verifies Eq. (17).
Effect of Phase Fraction
Traditional Approach

There have been many studies on the effect of phase fraction in turbulent flow for drop size determined by inertial subrange eddies (L >> d_{32} >> \eta):

\[
d_{32} / D = C_1 (1 + b \phi) \, \text{We}^{-3/5}
\]

non-coalescing: \( b = 3 \) (Doulah, 1975)
coalescing: \( 3 < b < 9 \)

- There is no systematic study of the effect of phase fraction on drop size for d_{32} below the Kolmogorov microscale
- Coulaloglou and Tavlarides (1977), Chesters (1991), and others have studied breakage and coalescence rates
Drop Size Dependence on Phase Fraction
Turbulent Flow – “Clean” Systems

• Strong effect of phase fraction on $d_{32}$

• Phase fraction effect attributed to:
  – Coalescence?
  – Turbulence suppression?

• Log-linear functionality:
  $d_{32}(\phi) = a \ln(\phi) + e$
  not
  $\frac{d_{32}(\phi)}{d_{32}(\phi = 0)} = 1 + b\phi$

• Conventional correlation would have initial slope $200 < b < 400$

Oil viscosity: 18.1 cP, no surfactant

Conventional correlation fails to correlate data for drops smaller than the Kolmogorov microscale
Drop Size Dependence on Phase Fraction

Laminar Flow – “Clean” Systems

- Same general trend for $d_{32}$ dependence on volume fraction as for turbulent flow
- Again log-linear:
  \[ d_{32}(\phi) = a \ln(\phi) + e \]
- Implies that the rate of coalescence is much greater when the flow is locally laminar, as is the case for both the laminar and turbulent flow data
Effect of Phase Fraction
Coalescence Fundamentals

For 2 drops of equal size, the coalescence rate is given by:

\[ \Psi(d,d) = C \times P \]

- \( C \) = specific collision rate (per unit volume) for 2 drops
- \( P \) = collision efficiency, or probability that the colliding drops will coalesce during their contact interval

Following Chesters (1991) (for simple shear flow), it can be shown that the collision efficiency approaches unity in laminar shear flow & sub-Kolmogorov turbulent flow.
High Coalescence Rate with a Viscous Continuous Phase ??

- Reservation: *High continuous phase viscosity should inhibit film drainage and thereby suppress coalescence*
- However: “In many pure liquid systems, drainage is controlled predominantly by the motion of the film surface.” (Chesters, 1991)

Low $\mu_d$ promotes high interface mobility
Dilute Surfactant Laden Systems

• Comprehensive static and dynamic pendant drop experiments
  – Surfactant diffusion coefficient and partition coefficient
  – Surface dilatational modulus (interfacial elasticity)

• Comprehensive dilute liquid-liquid dispersion experiments
  – DSD data: silicone oil - aqueous surfactant systems
    “clean” silicone oil - aqueous methanol systems
  – Silverson L4R mixer with slotted stator head

• Mechanistic theories
  – Models for mean drop size now account for interfacial elasticity
    ➢ Modified ‘Calabrese’ correlations and ‘spring & dashpot’ models

• Much learned but several data interpretation issues remain

• More controlled experiments have provided additional insight
  – Breakup of a laminar, surfactant laden liquid-liquid jet

• Some experimental results for more concentrated systems
  – Water in oil dispersions in the same Silverson L4R mixer
High Speed Photography
10 cP Silicone Oil Jet into Aqueous Solution

Movies recorded at 3,000 fps
Playback at 30 fps
Time lapse: 1% of real time

Jet optically inverted for post processing

Jet direction

Deionized Water

Full Jets

1xCMC Tergitol TMN-6

Breakup Zone
Dilute Surfactant Laden Systems

Silverson Model L4R Rotor-Stator Mixer w/slotted stator head

Disperse silicone oils in aqueous surfactant solutions
Interfacial Phenomena

Predict Surface Dilational Modulus due to Marangoni Stress

\[ E^{SD} = \frac{R \ T \ \Gamma_\infty \ \left( \frac{C}{a_L} \right)}{1 + \sqrt{D_S \ t_{def}} \left( \frac{a_L}{\Gamma_\infty} \right) \left( \frac{C}{a_L} + 1 \right)^2} \]

Surfactant Bulk Concentration, mol/l

Surfactant Dilational Modulus, mN/m

\[ \mu_d = 100 \text{ cP} \]
\[ d = 10 \mu m \]
\[ \varepsilon = 588 \text{ W/kg (5,000 rpm)} \]

Diffusion controlled adsorption

CMC ↓
Interfacial Phenomena Experiments
Silicone Oils in Aqueous Surfactant Solutions

Fit Equilibrium Data to obtain CMC, $\Gamma_\infty$ & $a_L$

<table>
<thead>
<tr>
<th>Surfactant</th>
<th>CMC (mol/l)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tergitol TMN-6</td>
<td>1.0x10^{-3}</td>
</tr>
<tr>
<td>Triton X-100</td>
<td>2.2x10^{-4}</td>
</tr>
<tr>
<td>Triton X-165</td>
<td>1.4x10^{-4}</td>
</tr>
</tbody>
</table>

\[
\sigma_{eq} = \sigma_0 - \Gamma_\infty RT \cdot \ln \left(1 + \frac{C}{a_L}\right)
\]

100 cP Silicone Oil
(Independent of viscosity grade)
Dynamic Interfacial Phenomena Experiments
Pendant Drop Method

Fit Surface Tension Data to get Surfactant Diffusion Coefficient

Ward –Tordai Equation

\[ \sigma(t)_{t\to\infty} = \sigma_{eq} + \frac{RT\Gamma_{eq}^2}{2C} \sqrt{\frac{\pi}{D_S t}} \]

Triton X-100
C = 1x10^{-5} \text{ mol/l}

D_S = 4.94x10^6 \text{ cm}^2/\text{s}

Dynamic surface tension of aqueous drops in air
Liquid-Liquid Dispersion Experiments

Effect of Surfactant Concentration on Drop Size

Maxima in drop size are attained in the same concentration range where the surface dilatational modulus peaks
Correlation for Mean Drop Size

Dispersed phase viscosity 10 to 100 cP

\[ \frac{d_{32}}{D} = 0.093(We \ N_{Re})^{-1/3} \left[ 1 + 24.44 \ Vi \ N_{Re}^{1/2} \left( \frac{d_{32}}{D} \right) \right]^{1/3} \]

\[ \mu_d^{\text{eff}} = \mu_d + 0.0032 E \ sd \ \frac{2t_{\text{def}}}{d_{32}} \]

RMSD = 21.5%

\[ \frac{d_{32}}{D} = 0.055 \ We^{-3/5} \left[ 1 + 2.06 \ Vi \left( \frac{d_{32}}{D} \right)^{1/3} \right]^{3/5} \]

\[ \mu_d^{\text{eff}} = \mu_d + 0.0035 E \ sd \ \frac{2t_{\text{def}}}{d_{32}} \]

RMSD = 28.5%

Correlations for ’clean systems’ can be modified to account for surface elasticity
Prediction and Measurement of Velocity Fields

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Prediction & Measurement of Velocity Fields

• Equation of Continuity: \( \nabla \cdot \mathbf{v} = 0 \)

• Navier Stokes Equations:
\[
\rho \left( \frac{\partial \mathbf{v}}{\partial t} + \mathbf{v} \cdot \nabla \mathbf{v} \right) = -\nabla P + \mu \nabla^2 \mathbf{v} + \rho \mathbf{g}
\]

• Reynolds Decomposition: \( \mathbf{v}_i = \overline{\mathbf{v}}_i + \mathbf{v}'_i \)
  – 3 components of velocity
  – Time averaging results in additional term known as Reynolds stress
    • Right hand side: \( -\nabla \cdot \rho \overline{\mathbf{v}'} \mathbf{v}' \)
RANS Equations
(Reynolds Averaged Navier Stokes Equations)

Ensemble Averaged Quantities

• Mean velocity: average
• RMS turbulent velocity: standard deviation
• Mean square turbulent velocity: variance
• Turbulent kinetic energy, $k$: $\frac{1}{2} \Sigma \text{variances}$
• Reynolds stress: covariance
• Energy dissipation rate, $\varepsilon$:
  – Frictional stress due all velocity gradients $\varepsilon = \nu \dot{\gamma}^2$
  – Total power (per mass) expended to overcome friction
  – Large spatial variation ($\varepsilon_{\text{avg}} \sim P/m$)
  – Proportional to turbulent kinetic energy: $\varepsilon \approx \frac{k^{3/2}}{\ell}$

\[ 2\gamma \nu \dot{\varepsilon} = 54 \]
Mean Velocity Field (normalized by tip speed) in Vertical Center-plane Containing the Shaft

Hybrid RANS Simulation with Fluent CFD Code

4,000 rpm

6,000 rpm
Mean Velocity Field (normalized by tip speed)

Hybrid RANS CFD Simulation (Fluent)
4,000 rpm

Time Periodic Region

Steady Region
Particle Tracking on Mean Velocity Field

4,000 rpm (one way coupling)

10,000 neutral density, 25 μm particles released just below mixing head
Stator slot: 6 x 10 x 12 mm

Rotor slot: 10 x 10 x 10 mm

Gap Width: 0.5 mm

Outer diameter of stator: 154 mm

12 rotor & 14 stator teeth

Pump fed turbulent water flow

IKA Prototype In-Line Rotor-Stator Mixer
IKA Prototype In-Line Rotor-Stator Mixer

CFD: RANS (with DPM) & LES
Measurements: LDA & PIV

12 rotor & 14 stator teeth
Outer diameter of stator: 154 mm

Rotor slot: 10 x 10 x 10 mm
Stator slot: 6 x 10 x 12 mm
Gap Width: 0.5 mm

Pump fed water flow
Mean velocity magnitude in stator slots 1 & 12 to 14

2-D RANS Simulation
330,000 cells
30 rps (1,800 rpm)
2.86 L/s (45.4 gpm)
Velocity and mass flow rate at stator slot exit plane
(illustrated with CFD generated mean velocity vectors)
3-D RANS Simulation & Comparison to LDA Data

LDA Data

30 rps, 2.55 L/s

U, m/s

3-D Simulation

No stator clearance

Angularly resolved mean velocities in stator slot 1

Rotation is now counter-clockwise
Angularly resolved turbulent kinetic energy in **stator slot 1**

TKE is somewhat under-predicted by 3-D simulation

---

**3-D RANS Simulation & Comparison to LDA Data**

**LDA Data**

- based only on \( r \& \theta \) components
- 30 rps,
- 2.55 L/s

**k, m^2/s^2**

**3-D Simulation**

- No stator clearance

---

~ 2.5 million cells
2-D Particle Image Velocimetry (PIV)

Radial (x) and tangential (y) velocity at various depths (z)

- Use pulsed lasers and camera to capture 2 images in rapid succession
- Discretize images into a grid of interrogation windows
- Observe local displacement of tracer particles
- Cross-correlation yields particle displacement and velocity
- Acquire instantaneous velocity fields in illuminated plane

Reynolds Number

\[ \text{Re} = \frac{U_{ss} D_{hs}}{\nu} \]

Shearing Number

\[ N_S = \frac{V_{Tip}}{U_{ss}} \]
0.84 L/s  
(13.3 gpm)  

\( N_S = 8.9 \)
\( Re = 6,211 \)

1.89 L/s  
(30 gpm)  

\( N_S = 3.9 \)
\( Re = 14,010 \)

Instantaneous Realizations

Mean

500 Realizations

10 rps (600 rpm),  \( z = 5.3 \) mm

Study No. 1
Flow Classification in Stator Slot 1

1. Mixing layer vortex
2. Recirculation region

$N_S = 11.5$
$Re = 9,573$

1.29 L/s, 20 rps
$z = 5 \text{ mm}$
Flow Classification in Stator Slot 1

3. Mixing layer

Mean Velocity Field

Study No. 2

\[ N_S = 11.5 \]
\[ Re = 9,573 \]

1.29 L/s, 20 rps
\[ z = 5 \text{ mm} \]
Flow Classification in Stator Slot 1

**Mean Velocity Field**

\[ z = 5 \text{ mm} \]

\[ N_S = 11.5 \]
\[ \text{Re} = 9,573 \]

4. **Rotor tip vortex**

\[ 1.29 \text{ L/s, 20 rps} \]
\[ z = 5 \text{ mm} \]

Study No. 2
Flow Classification in Stator Slot 1

5. Tooth boundary layer

6. Stator slot radial jet

$N_S = 11.5$

$Re = 9,573$

Mean Velocity Field

Study No. 2

1.29 L/s, 20 rps

$z = 5$ mm
Study No. 1

Instantaneous Velocity Field

Shear Layer Development

Structures are not dragged onto downstream stator tooth at low Shearing number
Magnitude of Deformation Rate, etc.

\[
\dot{\gamma} = \sqrt{\frac{1}{2} \Delta_{ij} \Delta_{ij}}
\]

Instantaneous deformation rate

\[
\bar{\dot{\gamma}} = \sqrt{\frac{1}{2} \bar{\Delta}_{ij} \bar{\Delta}_{ij}}
\]

Deformation rate based on mean velocity

\[
\dot{\gamma}' = \sqrt{\frac{1}{2} \Delta'_{ij} \Delta'_{ij}}
\]

Deformation rate based on fluctuations

Nominal Shear Rate:

\[
\dot{\gamma}_{nom} = \frac{V_{Tip}}{\delta}
\]

(0.5 mm shear gap)

Total energy dissipation rate:

\[
\varepsilon_{total} = \nu \dot{\gamma}^2
\]

Turbulent energy dissipation rate:

\[
\varepsilon_{turb} = \nu \dot{\gamma}'^2
\]

“Mean” deformation rate:

\[
\varepsilon_{mean} = \nu \bar{\dot{\gamma}}^2
\]

\[
V_i = \bar{V}_i + V'_i
\]

\[
\Delta_{ij} = \frac{\partial V_i}{\partial x_j} + \frac{\partial V_j}{\partial x_i}
\]
2-D Deformation Rate Magnitude ($s^{-1}$) Based on Instantaneous Velocity Gradients Estimated from PIV Data

10 rps, $N_S = 5.7$
\[ \dot{\gamma}_{nom} \approx 9,250 \, s^{-1} \]

1.29 L/s, $Re = 9,753$

20 rps, $N_S = 11.5$
\[ \dot{\gamma}_{nom} \approx 18,500 \, s^{-1} \]

Study No. 2

Average over 500 realizations of instantaneous deformation rate at $z = 5 \, \text{mm}$

Animate
2-D Deformation Rate Magnitude (s\(^{-1}\)) from PIV Data

10 rps, \(N_S = 5.7\), \(\dot{\gamma}_{nom} \approx 9,250\) s\(^{-1}\)

20 rps, \(N_S = 11.5\), \(\dot{\gamma}_{nom} \approx 18,500\) s\(^{-1}\)
RANS Simulation of Deformation Rate History
Particle paths and statistics via DPM

Criterion 1: particles that leave slot 1

Criterion 5: particles that leave slot 5

Slot 1

Slot 5

Animate
Maximum Deformation Rate Along Particle Path

\[ \dot{\gamma}_{\text{nom}} = 26,580 \text{ s}^{-1} \]

33.4 % experience max shear greater than nominal shear rate

\[ \frac{\dot{\gamma}_{\text{max}}}{\dot{\gamma}_{\text{nom}}} \]

Criterion 1

44.5% experience max shear greater than nominal shear rate

\[ \frac{\dot{\gamma}_{\text{max}}}{\dot{\gamma}_{\text{nom}}} \]

Criterion 5
IKA Prototype - Current Focus

- Evaluation of mesh quality and near wall treatment

- Compare PIV data to CFD simulations using refined computational grids and numerical techniques
Silverson In-Line Mixer Studies: CFD and Single Pass Drop Size Distribution

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BHR Group DOMINO Project Meetings
23 September 2015
Silverson L4R w/In-Line Attachment

Measurement of single pass DSD
Slotted Stator Head

Water Flow Rate: $0.5 < Q_v < 4$ L/min
$4,000 < N < 9,000$ rpm
$\mu_d = 0.9, 10, 60, 90, 540$ cP
Instantaneous Drop Count Frequency

\[ Q_v = 2 \text{ L/min} \]
Normalized Time Integrated (up to t) Sauter Mean Diameter

\[ Q_v = 2 \text{ L/min} \]
Dependence of $d_{32}$ & $d_{10}$ on $N$ and $Q_v$ for

$60$ cP

Lines of constant $N$

Lines of constant $Q_v$

Rotor speed $\rightarrow$

Volumetric flow rate $\rightarrow$

$d_{32} = \text{open symbols}$

$d_{10} = \text{closed symbols}$

60 cP Dispersed Phase Viscosity
Data Correlation and Analysis
Conventional Measures Fail to Correlate Data

• Equilibrium scaling
  – Energy dissipation rate:
    \[ \varepsilon = \frac{P}{\rho V_{MH}} \]

• Kinetic Scaling
  – Specific energy:
    \[ SE = \frac{P}{\rho Q_v} \]

• Rotor-Stator Performance Measure
  – Shearing number:
    \[ N_S = \frac{V_{Tip}}{Q_v \sqrt{nA_s}} \sim \frac{N}{Q_v} \]

Power draw:
\[ P = N_P \rho N^3 L^5 - Q_v \Delta P \]
CFD: RANS Simulation and Particle Tracking

Water: 1.0 L/min
5,000 rpm
25° C

Mean Velocity Field in Midplane

Animate
Cumulative Residence Time Distribution

\[ \tau = \frac{t}{\bar{\tau}} \]

Particle Tracking - BBO Equation

\[
\frac{d^2 X_p}{dt^2} = \frac{3 \mu \text{Re} C_D}{4 d_p^2 \rho_p} \left( \frac{d X_p}{dt} - \bar{V}_f \right) + \frac{\rho_p - \rho_f - g}{\rho_p} + \frac{1}{2 \rho_p} \left( \frac{DV_f}{Dt} - \frac{d^2 X_p}{dt^2} \right) + \frac{\rho_f}{\rho_p} \frac{DV_f}{Dt}
\]

- Drag
- Buoyancy
- Added Mass
- Pressure and viscous
Particle Tracking
Visualization of Difference in Particle Paths
Between Bubbles and Drops

50 µm diameter

Toluene Drops
Gas Bubbles

2,000 rpm
1.0 L/min
Water

Animate
Mean Velocity in Midplane of a Slot - Laminar Flow

Flow into / out of stator slot is either all discharge or all entrainment at any give time for low $N_{Re}$

$2,000$ rpm
$2.67$ L/min
$\mu = 5$ Pa·s
### Sliding Mesh RANS Simulations on 3 Scales

<table>
<thead>
<tr>
<th>Scale</th>
<th>Rotor Diameter</th>
<th>Rotor Slots</th>
<th>Stator Inside Diameter</th>
<th>Aspect Ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td>L4R</td>
<td>1.23 in</td>
<td>104 Slots</td>
<td>1.24 in</td>
<td>0.6</td>
</tr>
<tr>
<td>450 LS</td>
<td>4.5 in</td>
<td>560 Slots</td>
<td>4.51 in</td>
<td>0.27</td>
</tr>
<tr>
<td>600 LS</td>
<td>6 in</td>
<td>980 Slots</td>
<td>6.01 in</td>
<td>0.24</td>
</tr>
</tbody>
</table>
Mean Velocity Field Normalized by Tip Speed

Free Pumping
<table>
<thead>
<tr>
<th>Pumping Rate per Slot (L/s)</th>
<th>Qᵥ (L/s)</th>
<th>Nq</th>
<th>Pumping Rate per Slot (L/s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>L4R</td>
<td>0.22</td>
<td>0.06</td>
<td>0.0021</td>
</tr>
<tr>
<td>450LS</td>
<td>6.9</td>
<td>0.13</td>
<td>0.012</td>
</tr>
<tr>
<td>600LS</td>
<td>13.9</td>
<td>0.15</td>
<td>0.014</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Power</th>
<th>NP</th>
<th>P</th>
<th>P/Slot</th>
<th>SE</th>
<th>SE/Slot</th>
<th>Sfreq</th>
</tr>
</thead>
<tbody>
<tr>
<td>L4R</td>
<td>2.3</td>
<td>164</td>
<td>1.576</td>
<td>757</td>
<td>7.178</td>
<td>8E+5</td>
</tr>
<tr>
<td>450LS</td>
<td>1.3</td>
<td>1075</td>
<td>1.937</td>
<td>157</td>
<td>0.281</td>
<td>6E+5</td>
</tr>
<tr>
<td>600LS</td>
<td>1.2</td>
<td>1845</td>
<td>1.883</td>
<td>133</td>
<td>0.136</td>
<td>8E+5</td>
</tr>
</tbody>
</table>
Power Draw & Single Pass DSD in an IKA Labor Pilot 2000/4

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BHR Group DOMINO Project Meetings
23 September 2015
Power Draw and Single Pass DSD in IKA Labor Pilot

- Data for 1, 2 & 3 stages with 3 mill heads
  - Medium, Fine & Ultrafine Generators
  - Torque meter for power draw
  - 150 cP immersion oil in water for DSD
  - Rotor speeds up to 8,000 rpm
  - Volumetric flow rate: 1 to 5 L/min
  - Fed by progressive cavity pump

Power draw data to allow informed analysis of mean drop size data.
### ‘Medium’ Generator

- **2 Rows of Teeth**
- **9 Slots**
- **Slot width:** 1.575 mm
- **Slot height:** 3.5 mm

<table>
<thead>
<tr>
<th>Rotor Speed (rps)</th>
<th>1st Row (Diam, m)</th>
<th>Outer Row (Dia, m)</th>
<th>1st Row ($V_{tip}$, m/s)</th>
<th>Outer Row ($V_{tip}$, m/s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>66.67</td>
<td>0.047</td>
<td>0.057</td>
<td>9.85</td>
<td>11.94</td>
</tr>
<tr>
<td>83.33</td>
<td>0.047</td>
<td>0.057</td>
<td>12.31</td>
<td>14.92</td>
</tr>
<tr>
<td>100</td>
<td>0.047</td>
<td>0.057</td>
<td>14.77</td>
<td>17.91</td>
</tr>
<tr>
<td>116.67</td>
<td>0.047</td>
<td>0.057</td>
<td>17.23</td>
<td>20.89</td>
</tr>
<tr>
<td>133.33</td>
<td>0.047</td>
<td>0.057</td>
<td>19.69</td>
<td>23.88</td>
</tr>
</tbody>
</table>
‘Fine’ Generator

3 Rows of Teeth

13 Slots

Slot width: 0.965 mm

Slot height: 3.5 mm

<table>
<thead>
<tr>
<th>Rotor speed</th>
<th>1st Row (Diam, m)</th>
<th>2nd Row (Dia, m)</th>
<th>Outer Row (Dia, m)</th>
<th>1st Row ($V_{\text{tip}}$, m/s)</th>
<th>2nd Row ($V_{\text{tip}}$, m/s)</th>
<th>Outer Row ($V_{\text{tip}}$, m/s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>66.67</td>
<td>0.037</td>
<td>0.047</td>
<td>0.057</td>
<td>7.75</td>
<td>9.84</td>
<td>11.94</td>
</tr>
<tr>
<td>83.33</td>
<td>0.037</td>
<td>0.047</td>
<td>0.057</td>
<td>9.67</td>
<td>12.30</td>
<td>14.92</td>
</tr>
<tr>
<td>100</td>
<td>0.037</td>
<td>0.047</td>
<td>0.057</td>
<td>11.63</td>
<td>14.77</td>
<td>17.91</td>
</tr>
<tr>
<td>116.67</td>
<td>0.037</td>
<td>0.047</td>
<td>0.057</td>
<td>13.56</td>
<td>17.23</td>
<td>20.90</td>
</tr>
<tr>
<td>133.33</td>
<td>0.037</td>
<td>0.047</td>
<td>0.057</td>
<td>15.50</td>
<td>19.69</td>
<td>23.88</td>
</tr>
</tbody>
</table>
**‘Ultrafine’ Generator**

4 Rows of Teeth

22 Slots

Slot width: 0.72 mm

Slot height: 3.5 mm

<table>
<thead>
<tr>
<th>Rotor Speed (N,rps)</th>
<th>1st Row (Dia, m)</th>
<th>2nd Row (Dia, m)</th>
<th>3rd Row (Dia, m)</th>
<th>Outer Row (Dia, m)</th>
<th>1st Row (Tip Speed, m/s)</th>
<th>2nd Row (Tip Speed, m/s)</th>
<th>3rd Row (Tip Speed, m/s)</th>
<th>Outer Row (Tip Speed, m/s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>66.67</td>
<td>0.0345</td>
<td>0.043</td>
<td>0.052</td>
<td>0.06</td>
<td>7.23</td>
<td>9.01</td>
<td>10.89</td>
<td>12.57</td>
</tr>
<tr>
<td>83.34</td>
<td>0.0345</td>
<td>0.043</td>
<td>0.052</td>
<td>0.06</td>
<td>9.03</td>
<td>11.23</td>
<td>13.61</td>
<td>15.71</td>
</tr>
<tr>
<td>100</td>
<td>0.0345</td>
<td>0.043</td>
<td>0.052</td>
<td>0.06</td>
<td>10.84</td>
<td>13.51</td>
<td>16.34</td>
<td>18.85</td>
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<tr>
<td>116.67</td>
<td>0.0345</td>
<td>0.043</td>
<td>0.052</td>
<td>0.06</td>
<td>12.64</td>
<td>15.76</td>
<td>19.06</td>
<td>21.99</td>
</tr>
<tr>
<td>133.33</td>
<td>0.0345</td>
<td>0.043</td>
<td>0.052</td>
<td>0.06</td>
<td>14.45</td>
<td>18.01</td>
<td>21.78</td>
<td>25.13</td>
</tr>
</tbody>
</table>
Power Measurements - Water Motivation & Approach

- Find relationship between Power number and Reynolds number
- Install Futek TRS600 (# FSH01995, range 2 Nm) torque sensor in-line with rotor shaft via purpose built housing
- Monitor temperature rise across mixer to back up torque measurements
- Measure flow work via differential pressure gage
- Fed via progressive cavity pump
Calculating Dissipated Power

Sample plots — similar ones can be made at all configurations

\( \eta \) can be calculated from the power dissipated in the fluid:

\[
P_{\text{diss}} = P_{\text{meas}} - P_{\text{blank}} - P_{\text{flow work}} + P_{\text{blank flow work}}
\]

A significant amount of power is spent by bearings, etc. (\( P_{\text{diss}} \sim P_{\text{blank}} \))

The flow work terms are small
Dissipated Power

- Calorimetric method is less accurate, but does provide order of magnitude validation
- More power dissipated for 2 stages than for 1
- 1 Ultrafine stage plot is about the same as for 2 medium stages
- More power is transmitted to the fluid as the teeth get finer
3 lpm is used as a representative flow rate since the power dissipation is roughly independent of flow rate.

- Power per stage is roughly constant for each generator type.
- Ultrafine stage dissipates more power than medium and fine stages.
Toward Drop Size Scaling

- Constant $N_p$ implies fully turbulent flow in mixer
- Drop size data are mostly in the constant power number region
- Drop size data via PDA are dilute: $\phi \rightarrow 0$

For turbulent flow, there are mechanistic correlations for equilibrium DSD with respect to the Kolmogorov microscale: $\eta = (v^3/\varepsilon)^{1/4}$

With an eye toward the swept-volume approach, the generator fluid volume is used - red color $\rightarrow$

Range of $\eta$ is 3.8 to 8.3 $\mu$m
Single Pass Drop Size Distribution Data

Same procedure as for L4R in-line unit
Mean Drop Size Data: 1 Stage
Data mostly fall within inertial subrange ($\eta << d << D$)

1 Stage: Medium Generator

1 Stage: Fine Generator

1 Stage: Ultrafine Generator

$$\eta = \left( \frac{\nu^3}{\varepsilon} \right)^{1/4}$$
Mean Drop Size Data: 3 Stages
Data mostly fall within inertial subrange ($\eta << d << D$)

3 Stage: Medium Generator

3 Stage: Fine Generator

3 Stage: Ultrafine Generator

$\eta = (v^3/\varepsilon)^{1/4}$
Time Averaged DSD - Fine Generator

1 FG - 5.0 L/min

2 FG - 5.0 L/min

Number

Volume

$P_n(d)$

$P_V(d)$

$d, \mu m$

$d, \mu m$
Instantaneous DSD and RTD

Similarity of Residence Time Distribution

2 Stage Fine Generator

![Graph showing Instantaneous DSD and RTD with drop number frequency on the y-axis and t/τ on the x-axis. Different lines represent various flow rates and rotational speeds.]
Instantaneous DSD and RTD

Similarity of Instantaneous DSD

2 Stage Fine Generator
IKA Labor Pilot Observations

• Time averaged drop size distribution:
  – In general $d_{32}$ decreases as we increase rotor speed and the number of rows/teeth per stage, and decrease throughput; until an “equilibrium like” regime is achieved.
  – Except for the large size tail, operating conditions have a more pronounced affect on the volume distribution than on the number distribution.

• Transient phenomena:
  – For a given geometry, the drop count frequency depends on throughput, but not on rotor speed. The RTD’s collapse when normalized with the mean residence time.
  – For a given geometry, the normalized cumulative $d_{32}(t)$ appear to be self similar. What is needed are mechanistic correlations for $d_{32,\text{avg}}$. 
Equilibrium Drop Size Scaling: Inertial Subrange

- At higher power input and lower flow rate, mean drop size data appear to be independent of flow rate (and residence time), indicating that equilibrium may have been achieved.
- Drop sizes are within inertial subrange ($\eta << d << D$).
- Drop size data via PDA are relatively dilute ($\phi \rightarrow 0$).
- Compare selected data to inertial subrange scaling for equilibrium conditions.
- The characteristic device diameter is not known, so analyze data via local energy dissipation rate rather than We, etc.
- $d_{32}$ is proportional to $d_{\text{max}}$: $d_{32} = 0.45 \ d_{\text{max}}$.
- The inviscid and very viscous drop scaling limits provide an equally good fit to the data ($\mu_d = 150$ cP): 
  \[ d_{32/\text{max}} \sim \frac{\sigma^{3/5}}{\rho_c^{3/5}} \varepsilon^{-2/5} \]
  \[ d_{32/\text{max}} \sim \frac{\mu_d^{3/4}}{(\rho_c \rho_d)^{3/8}} \varepsilon^{-1/4} \]

Inviscid drops

Very viscous drops
Comparison with Davies Plot

- Davies (1987) - drop size can be correlated to local (maximum) energy dissipation rate independent of device.
- Bottom plot shows only data whose drop sizes were mostly independent of flow rate: $\Delta (3 \text{ lpm to 5 lmp}) < 10\%$
- Bottom plot is used to estimate $\varepsilon_{\text{max}}$.
The energy dissipation rate used previously was based on total the fluid volume around the mill heads, $\varepsilon_{\text{avg}}$.

If equilibrium has been achieved, the data show that $\varepsilon_{\text{max}} \approx 9 \varepsilon_{\text{avg}}$, or the high shear region is approximately $1/9$ of the fluid volume.

$Davies\ Plot: \quad d_{\text{max}} = 1010\varepsilon^{-0.384}$
• The power put into pumping is significantly smaller than the power expended by viscous dissipation
• The Power draw is independent of flow rate
• Means drop size correlates slightly better with “standard” inertial subrange scaling than with high drop viscosity inertial subrange scaling (assuming there is sufficient residence time to reach equilibrium)
• There is correlation between data whose drop sizes are less affected by flowrate and data which follow the Davies plot - i.e. inertial subrange scaling: $d_{\text{max}} \sim \varepsilon^{2/5}$
• Based on equilibrium drop size: $\varepsilon_{\text{max}} \approx 9 \varepsilon_{\text{avg}}$
• Much more could be done
Crystal Wet Milling Studies in Silverson L4R In-line Lab Scale Mixer

Establish mechanistic basis for crystal wet milling
Mechanisms for High Shear Wet Milling
Experiments & Simulation

• Objectives:
  – Mechanistic understanding of crystal fragmentation in high shear environments
  – Simulation tools for scale up of crystal wet milling processes

• Experiments in Silverson L4R inline mixer (square hole head)
  – Parametric study - breakage/fracture mechanisms (PBE kernels)
    • Mill operating conditions (rotor speed, throughput & stator geometry)
    • Crystal mechanical properties (shape, hardness & stress intensity factor)
    • Slurry concentration

• Simulation tools
  – Population balance kernels
  – Sliding mesh Fluent simulations
  – gPROMS multi-zonal population balance model

• Experimental program on solid ground
  – Extensive use of Horiba LA-950 Particle Size Analyzer
    • Systematic error analysis of measured PSD
  – Lasentec D600L FBRM probe recently acquired
Crystal Wet Milling Program

Fluent CFD
Flow & Deformation Fields

Zone Definition

PSE gPROMS
Multi-Zonal PBE

PBE Equations
Breakage Kernels

Silverson L4R, 450LS, 600LS
In-line Units

Silverson L4R Batch Unit

IKA Prototype In-line Unit

IKA Labor Pilot In-line Unit

Silverson L4R In-Line Unit

Crystal Wet Milling
Experiments

Model Validation

Literature Models,
Mechanistic Model Development

IKA Labor Pilot In-line Unit
Liquid-Liquid Dispersion Program

Fluent CFD
Flow & Deformation Fields

Zone Definition

PSE gPROMS Multi-Zonal PBE

PBE Equations Breakage Kernels

Model Validation

Drop Breakup Experiments

Silverson L4R, 450LS, 600LS In-line Units
Silverson L4R Batch Unit
IKA Prototype In-line Unit
IKA Labor Pilot In-line Unit

IKA Labor Pilot In-line Unit
Silverson L4R In-Line Unit

Silverson L4R Batch Unit
Rushton Turbine Stirred Tank

Literature Models, Mechanistic Model Development