Impeller Design for Liquid-Liquid Dispersion Using VisiMix RSD/Turbulent

Matthew Jörgensen

matt.jorgensen@nalasengineering.com

860-460-9611

Jerry Salan

jerry.salan@nalasengineering.com

Available for public release

Outline

- Modeling a liquid-liquid system in VisiMix
  - How we used PVM to calculate drop size
  - PVM drop size matched to VisiMix to "calibrate" the model
  - RSD to model disperserator
  - Mapped VisimixRSD properties to VisiMixTurbulent to model drop size
  - Used VisiMix to evaluate multiple impeller configurations

What do we do?

NALAS Engineering Services, Inc.

- Transition ***chemical processes*** to the plant environment
  - Identify engineering challenges including heat transfer, mass transfer, and mixing
  - Evaluate chemistry in the laboratory using *in situ* tools (IR, Raman, FBRM, PVM, heat flow)
- Evaluate pilot and production equipment. Validate processes through scale-down experiments
- Develop low-cost chemical *processes*

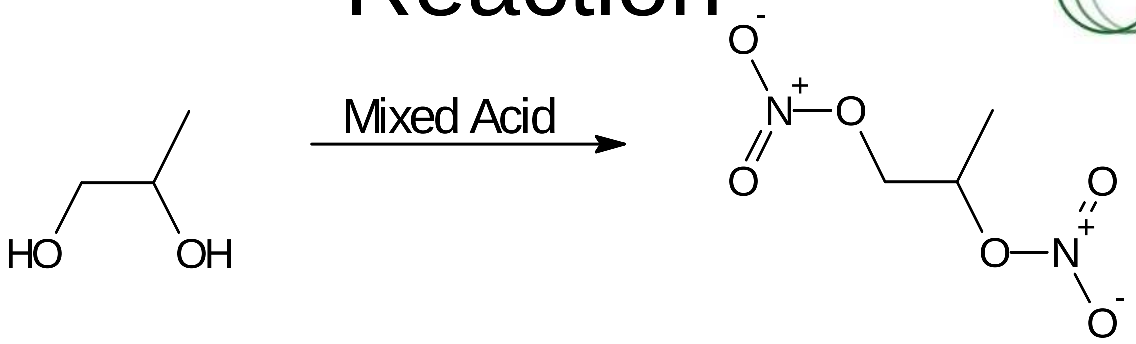
Background

- Design an automated laboratory reactor to replace the current lab system for the evaluation of raw materials in the production of Propylene Glycol Dinitrate (PGDN).
- Maintain same degree of mixing as traditional system

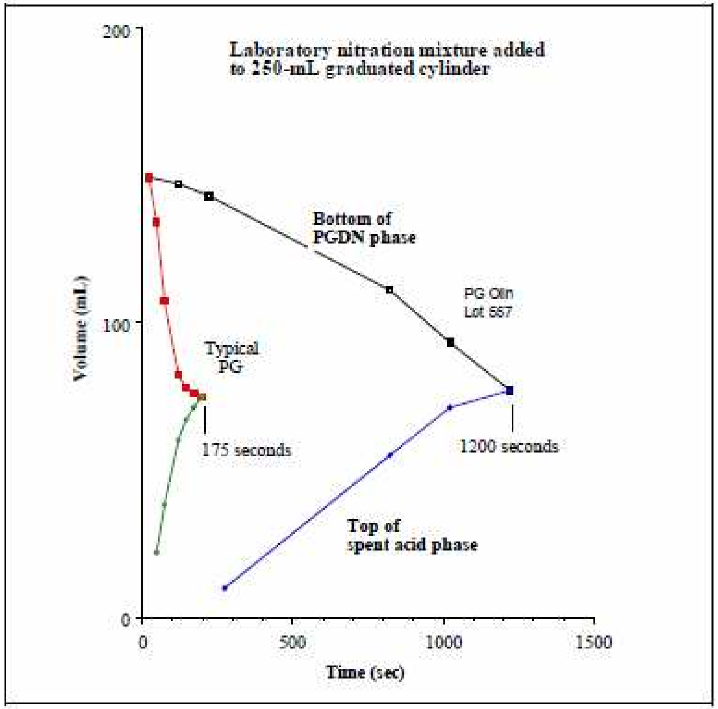
Background

- PGDN is the main ingredient in Otto II fuel used in torpedoes.
- It is manufactured in a continuous process
- The nitration is highly exothermic and requires intimate mixing to avoid 'hot spots'
- Poorly mixed systems can result in 'fume offs'

Reaction



- Propylene Glycol is added to mixed acid (nitric and sulfuric)
- Resulting liquid PGDN is the *light phase* suspended in the *heavy phase* mixed acid
- In the past, propylene glycol shipments have been contaminated with small amounts of impurity resulting in poor separation in production equipment
- Lab-scale nitration was designed to mimic same degree of mixing as production nitrator
- Each shipment must meet a specification, Including separation time, before being used in the plant.



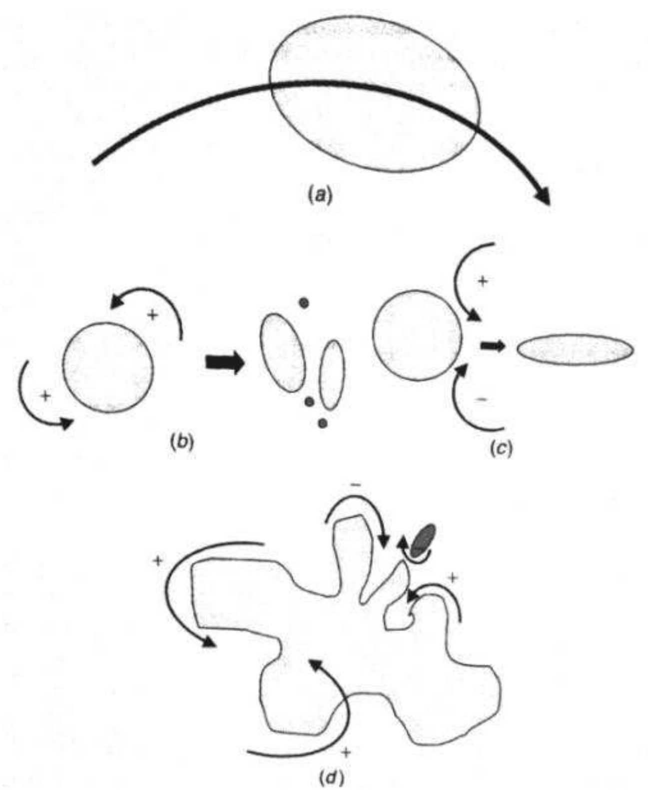
Laboratory Reactor Constraints

- The main point of the automation is to increase worker safety, while maintaining same degree of mixing
  - Allow for comparison back to historical data
  - Droplet size may impact separation times
  - Identify problematic lots of propylene glycol
- Match the mixing that they have in the current setup
  - VisiMix to model both existing and proposed lab reactor

Liquid-Liquid
Drop Formation in Turbulent Flow

Convection if eddies are larger than drop

Erosion by co-rotating eddies



Elongation by counter-rotating eddies

Overlay of multiple scales of turbulent deformation

Liquid-Liquid
Drop Formation in Turbulent Flow

In a low viscosity dispersed phase, the classical relationship between mean diameter and impeller Weber number:

$$\frac{d_{32}}{D} \propto We^{-0.6} \quad \text{with} \quad We = \frac{\rho N^2 D^3}{\sigma}$$

$$\gamma \propto Tip\ Speed\ (= \pi N D)$$

d_{32} = Sauter mean drop diameter
D = impeller diameter
σ = Interfacial tension
ρ = Density
N = impeller speed
γ = shear rate

It is possible to match drop diameter and level of dispersion between two geometrically similar systems that use the same dispersed phases by matching the shear in the two systems.

Simulant Testing

NALAS Engineering Services, Inc.

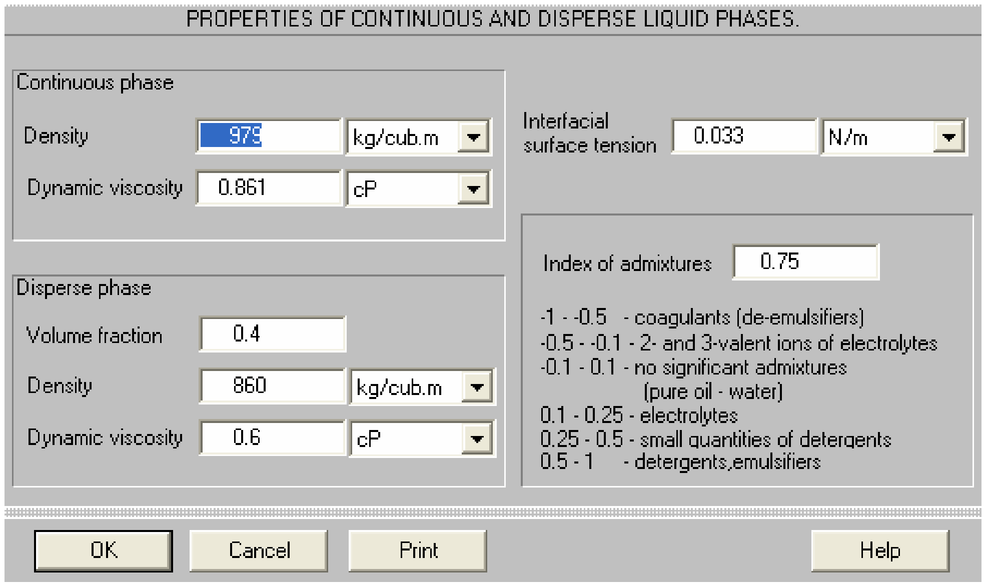
- Test system was Toluene/water.
- Direct comparison of the 'existing' laboratory system vs. the 'proposed' laboratory system

Existing Setup
"Disperserator"

Proposed Setup
Traditional Impellers

VisiMix Inputs for Liquid-Liquid Mixing

- Interfacial Surface tension between the two phases
- Density of both phases
- Index of admixtures
  - This is a measure of the system to stabilize drops
    - Electrolytes
    - Surfactants
    - Etc.



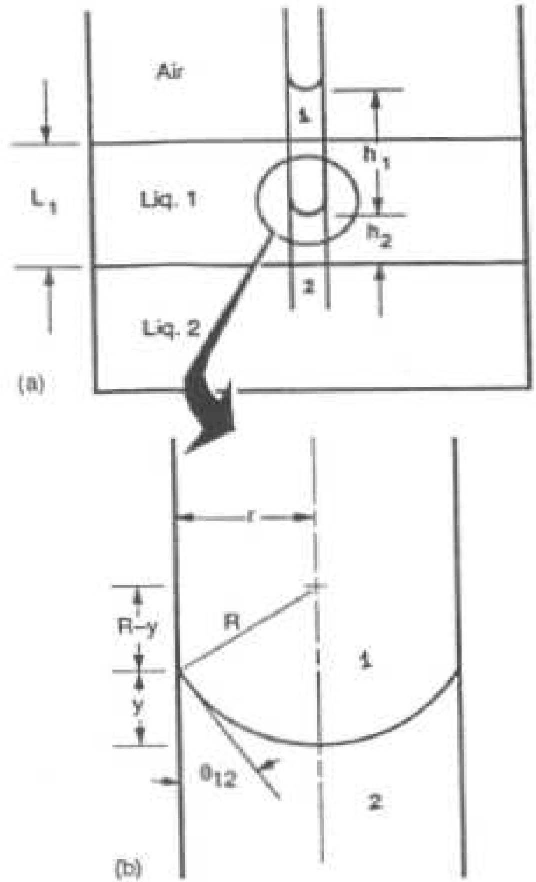
Required Inputs

Interfacial tension

$$\sigma_{12} = -\sigma_{1a}\frac{\cos\theta_{1a}}{\cos\theta_{12}} + \frac{gr}{2\cos\theta_{12}}(\rho_1 h_1 + \rho_2 h_2 - \rho_1 L_1)$$

Were :

- σ_{12} =interfacial tension between the two liquids
- σ_{1a} =surface tension of the light phase
- θ_{12} =angel of contact of the liquid-liquid meniscus with the capillary wall
- θ_{1a} =angel of contact of the light phase meniscus with the capillary wall
- g = acceleration due to gravity
- r = radius of the capillary
- ρ1 and ρ2 =densities of the respective phase.
- h_1, h_2, and L_1 are measurements taken as shown in figure



Required Inputs

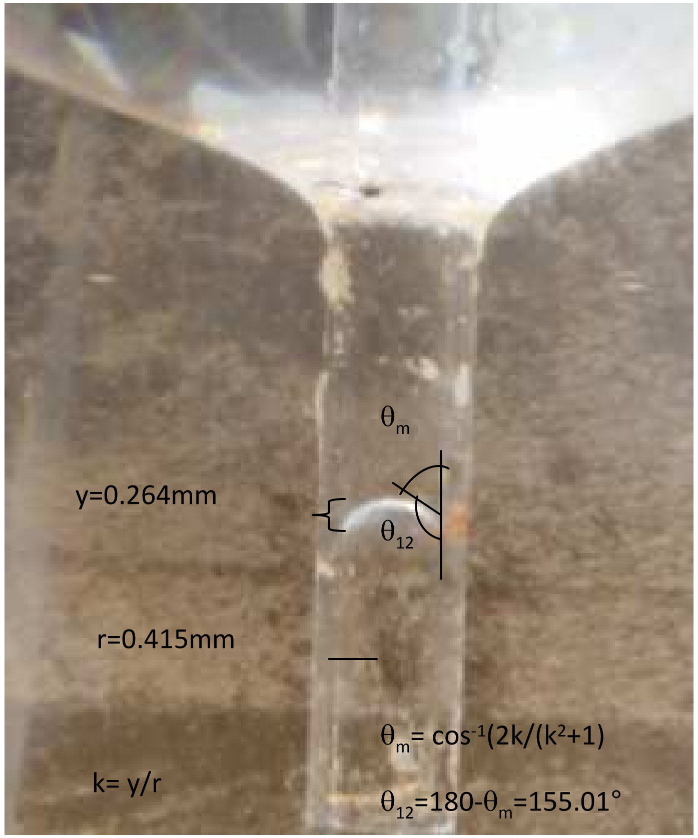
NALAS Engineering Services, Inc.

- Densities of the two phases were measured after the phases had been mixed and allowed to separate.
- This is to account for the change in density due to the solubility of the two materials with each other.

Required Inputs

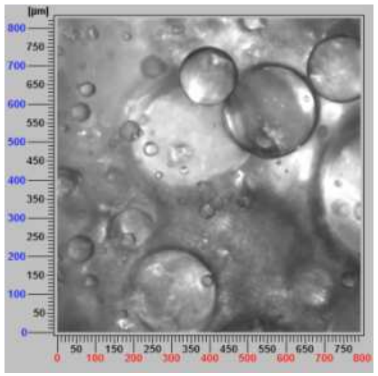
$$\sigma_{12} = -\sigma_{1a}\frac{\cos\theta_{1a}}{\cos\theta_{12}} + \frac{gr}{2\cos\theta_{12}}(\rho_1 h_1 + \rho_2 h_2 - \rho_1 L_1)$$

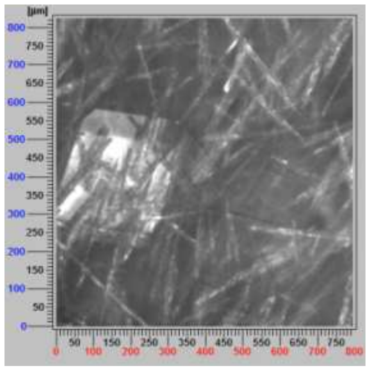
- Photograph of Toluene/water interface
- Measured interfacial tension our system (Toluene/Water)
  - 0.0327 $N \cdot m^{-1}$
- Reported/reference interfacial tension for Toluene/Water
  - 0.0364 $N \cdot m^{-1}$.

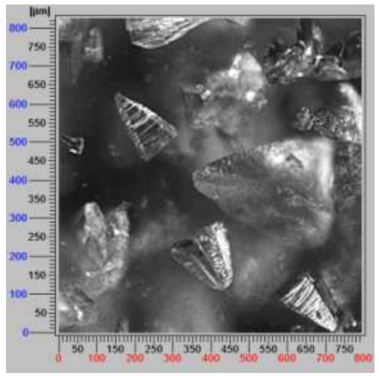


Particle Vision Microscopy: PVM

In situ probe that allows for:
- Detect multiple phases: Gas, Bubbles, Droplets, Oil
- Characterize Particle Shape
- Polymorphic crystallization characterization
    - Visualize morphology changes
    - Understand dynamics of polymorph transitions
- Characterize surface roughness
- Understand particle dynamics and interactions: growth, nucleation, agglomeration, and breakage phenomena
- Determine root cause of particle processing problems

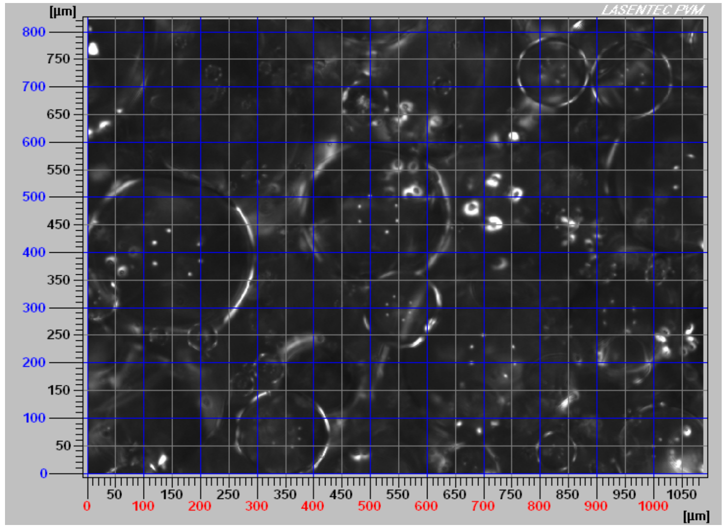






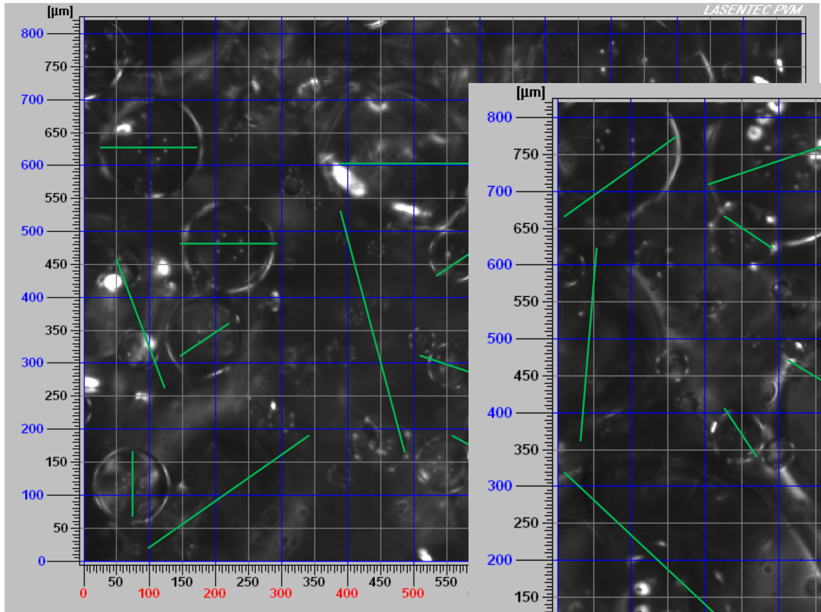
Validate Model Using PVM

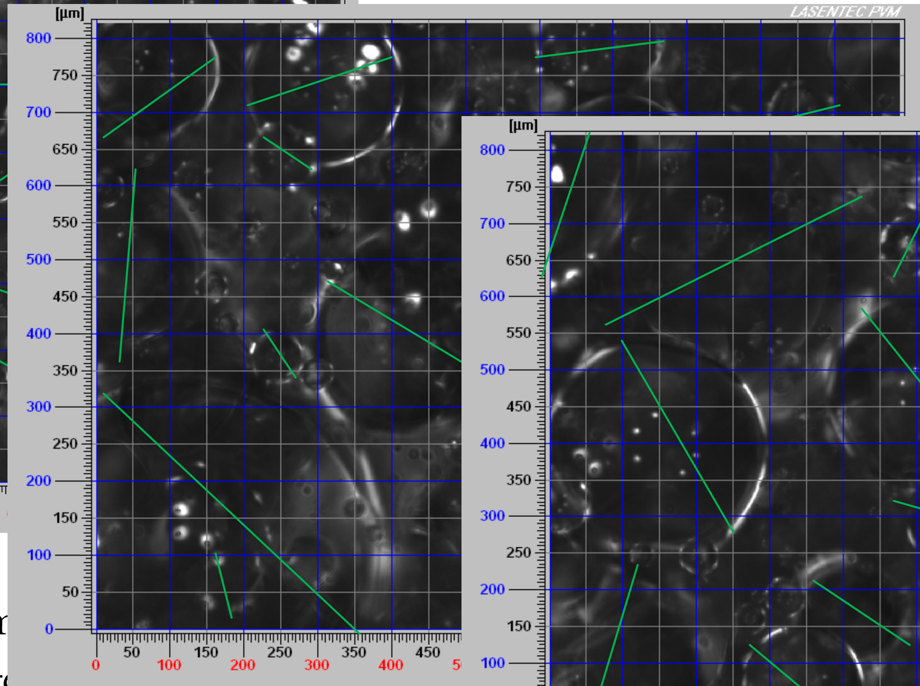
- Taking the PVM data at one setup to test the model for the admixture value.
- Comparing drop size distribution to the VisiMix values
- By matching the shear between systems we hope to match drop size, surface area, and mixing.
  - Mean drop size

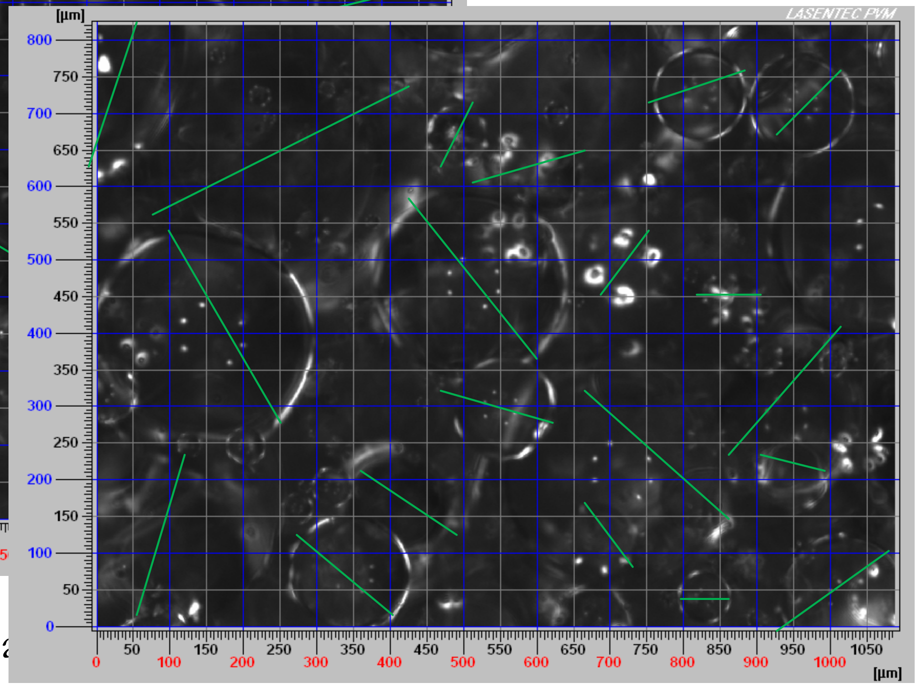


Calculating Drop Diameter from PVM

Nalas Engineering Services, Inc.







Average of drop diam… PVM image

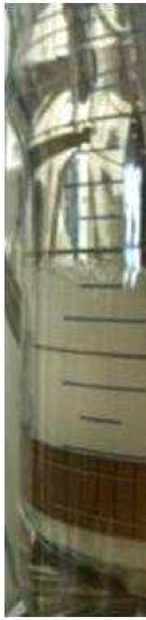
Repeat with a…

Repeat a third time…

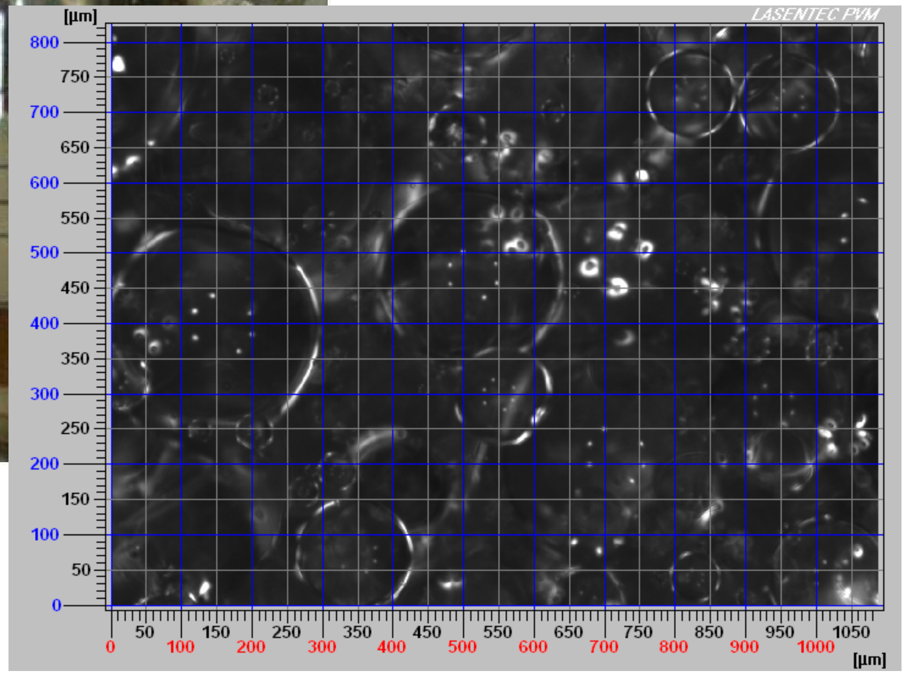
The average diameter for all three images is then averaged again and that value is the drop diameter for that RPM

RC-1 Experiments

- Pitch blade impeller with PVM and Tr as baffles.

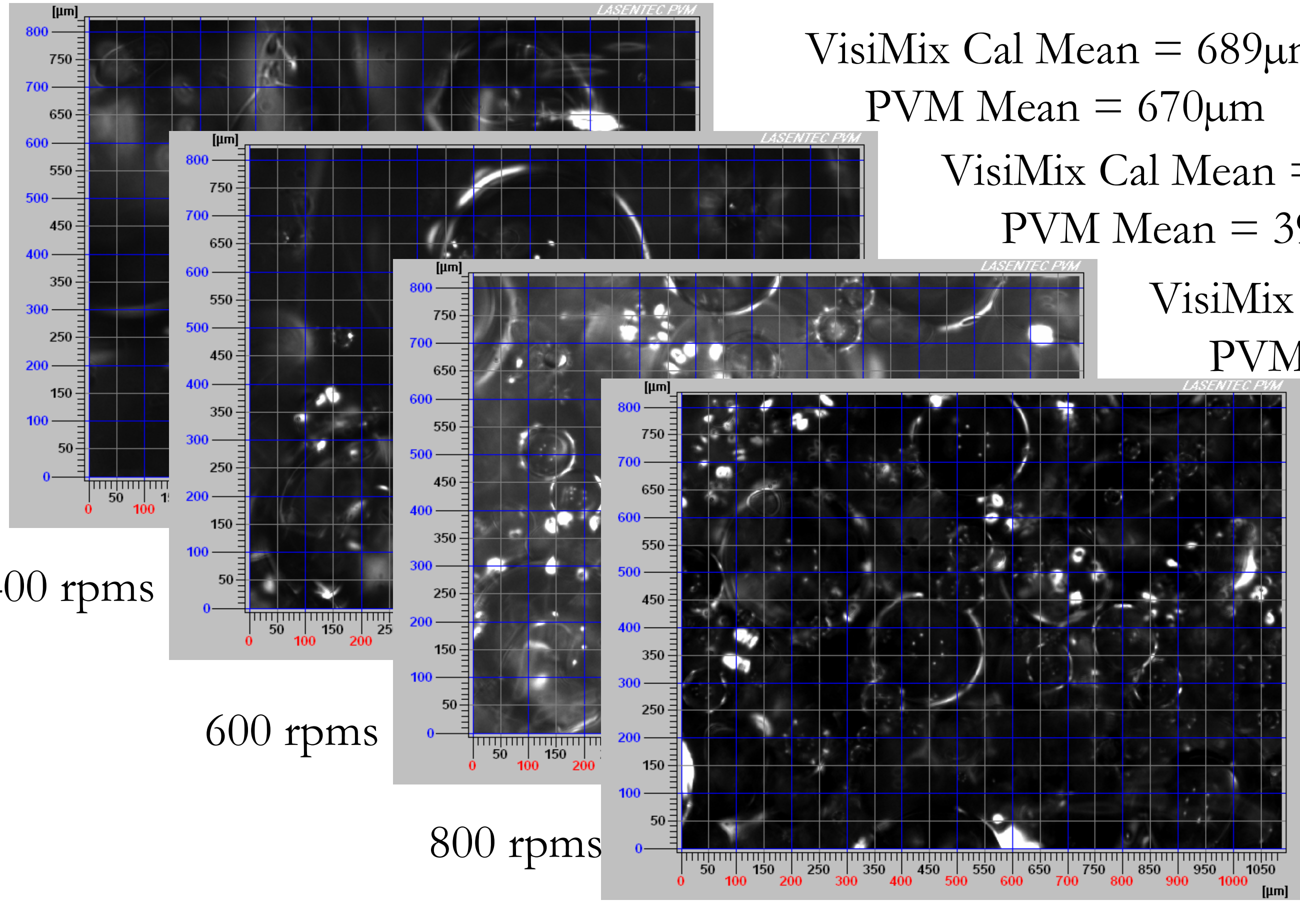


PVM mean ≈ 280 μm



VisiMix calculated mean = 282 μm
with admixture value set to 0.75

RC-1 Experiments Using PB-Impeller



VisiMix Cal Mean = 689μm
PVM Mean = 670μm

VisiMix Cal Mean = 403μm
PVM Mean = 397μm

VisiMix Cal Mean = 314μm
PVM Mean = 301μm

VisiMix Cal Mean = 281μm
PVM Mean = 275μm

400 rpms

600 rpms

800 rpms

10000 rpms

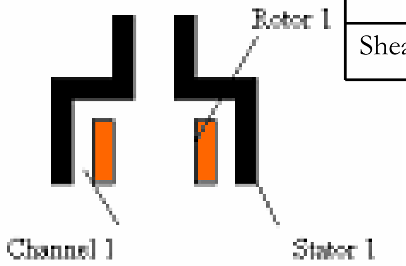
VisiMix RSD

VisiMix RSD enables you to quickly calculate—

- Shear rates and stresses in internal spaces of the High Shear Mixer
- Pumping capacities
- Power consumption and torque

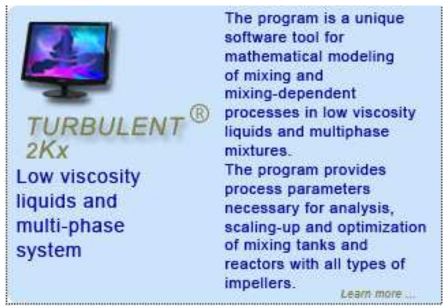
Modeling

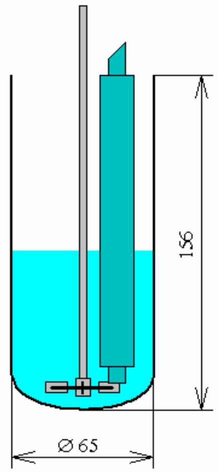
- VisiMix models both traditional type impellers (Turbulent 2K) and rotor stator mixers (RSD)
- First calculate mixing parameters using rotor stator model
- Match the output using Turbulent 2K
  - Trial and error by simply changing rpm



RPMS	4000
Shear Rate [1/sec]	30800
Shear Stress [N/sq.m]	54.6

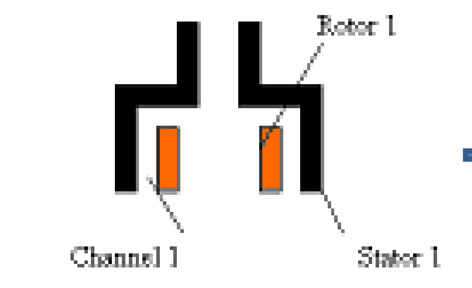
RPMS	1860
Shear Rate [1/sec]	30800
Shear Stress [N/sq.m]	54.6

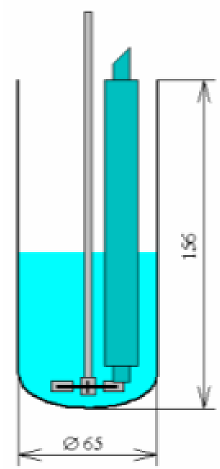




Disperserator Experiments

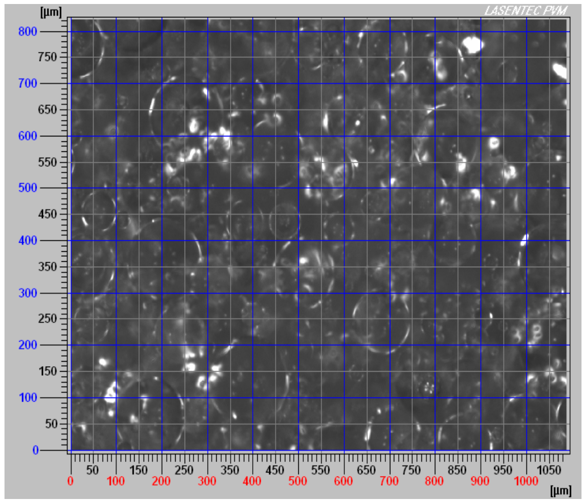
Modeling:





Mean diameter
=138 μm

Experimentation:



Mean diameter
≈ 130 μm

Nalas Engineering Services, Inc.

Modeling Configurations in RC-1 as Compared to the Disperserator

Configuration	Baffles	Mean drop size (µm)	Max shear rate (1/sec)	Shear Stress Near Impeller (N/sq.m)
RC-1 with PB impeller at 1000 rpms	PVM baffle (experimental set up)	281	30800	54.6
RC-1 with PB impeller at 1000 rpms	4 flat blade baffles	259	7220	12.8
RC-1 with PB impeller at 2000 rpms*	4 flat blade baffles	171	20500	36.2
RC-1 with 6 blade RDT impeller at 1000 rpms	4 flat blade baffles	220	11000	19.6
RC-1 with 6 blade RDT impeller at 2000 rpms*	4 flat blade baffles	142	31300	55.4
Disperserator at 4000 rpms	PVM baffle (experimental set up)	138	30800	54.6

**note: predicted value is higher than maximum system rpms for this particular impeller*

What we learned from VisiMix about the Reactor Design

- The current RC-1 configuration with a pitched blade impeller does not provide sufficient mixing when compared to the existing rotor/stator system (to be expected).
- The addition of a RDT gives large improvement as compared to the Pitched Blade Impeller, but with a 1000 rpm limit on the automated reactor system, still cannot match the dispersator.
- If the rpms limit can be increased then it may be possible to use a RDT in the final configuration.
    - If not then a disperserator will have to be utilized.

NALAS Engineering Services, Inc.

VisiMix Model & Mettler Toledo Tools: ***Scaling-up with Confidence***

- Utilization of the *in-situ* PVM allowed us to validate the VisiMix model giving us confidence in the results and experimental methodology
- VisiMix model provided accurate prediction of mixing parameters for various configurations
- Mettler Toledo tools allowed us to quickly validate the VisiMix models and understand the design aspects of the proposed system
  - Proposed system will not provide degree of mixing required without some modifications

Conclusions

- VisiMix accurately predicts mixing parameters for both traditional impellers and rotor/stator systems for liquid-liquid mixing
- By modeling the dispersion in the historical laboratory equipment we are able to identify automated reactor configurations that will maintain the same degree of mixing.

Acknowledgments

- Mr. Chuck Painter, Director, Navy ManTech Office
- Mr. Patrick Skahan , NSWC Indian Head Division
- Mr. Tod Ricks , NSWC Indian Head Division
- Mrs. Shilpa Amato, NSWC Indian Head Division
- Mr. Jerry Salan and Ms. Shannon Lenahan, Nalas Engineering