Rotor **S**tator **D**ispersers and **E**mulsifiers

Moshe Bentolila

VisiMix Ltd,
PO Box 45170,
Jerusalem, 91450,
Israel
Tel: 972 - 2 - 5870123
Fax: 972 - 2 - 5870206
E-mail: info@visimix.com

Contents

- Introduction
- VisiMix Mathematical Model for Emulsification
- Applications
- Customers Example
  - ✓ Impeller Design for Liquid-Liquid Dispersion Using VisiMix RSD/Turbulent
  - ✓ Selectivity Improvement in Homogeneous Reaction

Introduction

About Us

VisiMix is a unique software enabling chemical engineers, process engineers and R&D personnel to visualize mixing processes via a simple, user friendly interface.

Our products allow significant savings in time and costs by drastically reducing the need for trial-and-error. They have been successfully adopted by hundreds of companies.

software for chemical engineering

Customers and Markets

BASF

Pfizer

MERCK

BÜCHI

TEVA

Dow®

GE Healthcare

3M

RANBAXY

De Dietrich

EVONIK INDUSTRIES

MeadJohnson Nutrition

HERCULES

L'ORÉAL

Lubrizol

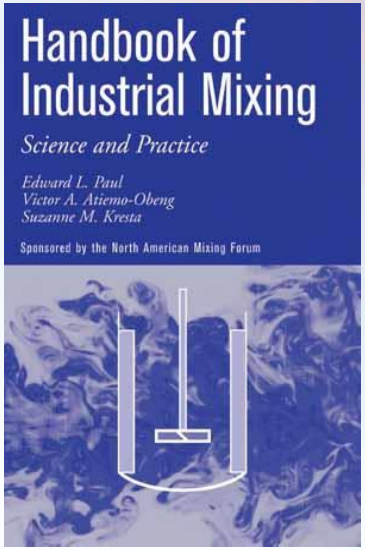
VisiMix

software for chemical engineering

What Our Customers Say About Us

From the Dow Chemicals Intranet Website

"VisiMix: This is a highly accessible PC software for mixing calculations available from VisiMix Ltd., an Israeli company. It is a rating calculation tool for both non-reactive and reactive mixing involving blending, solid suspension, gas dispersion, liquid-liquid dispersion, or heat transfer processes in stirred vessels. It calculates the important process parameters for single- and two-phase systems – power consumption, circulation rates, local concentrations of solutes and suspended particles, drop size, concentrations of reactants, etc … "



Dr. Victor Atiemo-Obeng

Dow Chemicals Co.

Customers and Markets

1.3M, USA
2.Afton, USA
3.Air Products, USA
4.Alkermes
5.AllessaChemie
6.ASEPCO
7.Ashland Hercules
8.BASF, USA
9.Belinka Belles
10.Celgene
11.Chemagis
12.DeDietrich
13.Dow chemical
14.Eni - Milan
15.Evonik Degussa
16.GE Technologies
17.GE Healthcare
18.Global Tungsten & Powders
19.Honeywell-UOP
20.Ineos Styrenics
21.Lubrizol
22.Merck - Schering-Plough
23.Mitsubishi
24.NALAS-Jerry Salan
25.Nan Ya Plastic
26.Nippon
27.Novartis
28.NRDC, India
29.Ocean
30.Pfizer
31.Polimeri
32.Praxair
33.Ranbaxy
34.Samsung, Korea
35.Sunovion Sepracor
36.SES
37.Solvay
38.Tecnicas Reunidas
39.Tecnimont, Italy
40.Teva Global
41.US Navy, USA
42.Alcon, USA
43.Arizona Chemical
44.JM Huber
45.Jotun
46.Lek
47.Matrix\Mylan
48.MJN - Mead Johnson Nutrition
49.Ranbaxy
50.R.C.Costello
51.Styron
52.Tami
53.Taro
54.Xellia
55.Ash Stevens Inc…….

.

Total > 200 customers

VisiMix Mathematical Model for Emulsification

VisiMix RSDE – Simulation of Emulsification in Rotor-Stator Dispersers

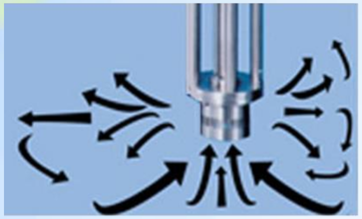
For the first time ever, you can model emulsification processes in dispersers!

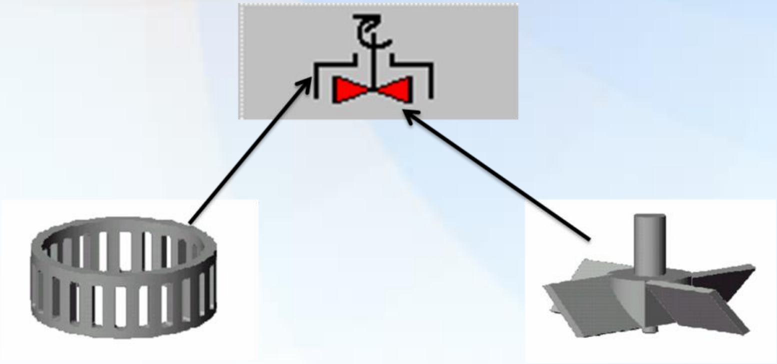
An enhanced version of VisiMix RSD which **extends RSD functions to calculations of emulsification**. Modeling and calculations both for Rotor/Stator Dispersers and Tooth Disk Dispersers Various external and internal mounting schemes – **stand-alone or combined with other mixing devices**. Various media and process configurations

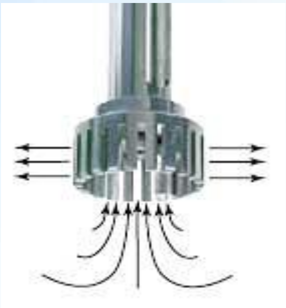
VisiMix RSDE offers all capabilities of VisiMix RSD and much more:

New enhanced capabilities in calculations of emulsification parameters for selecting and operating emulsification equipment

- **Mean drop size**
- **Drop size distribution**
- **Emulsification dynamics, etc**







Evaluation of **main operational characteristics of RSD devices** (shear rates, specific power, pumping capacity, etc.) Modeling of batch and continuous flow homogenization for RSD devices as function of **the equipment design and media properties for Newtonian and non-Newtonian media** Based on unique mathematical models, tested and verified 5 years of experimental research and algorithm development in a dedicated laboratory.

The New RSDE continues to calculate the main hydrodynamics and mixing parameters from the previous versions named - RSD for instance:

1) Evaluation of the main operational characteristics of the RSD devices such as –

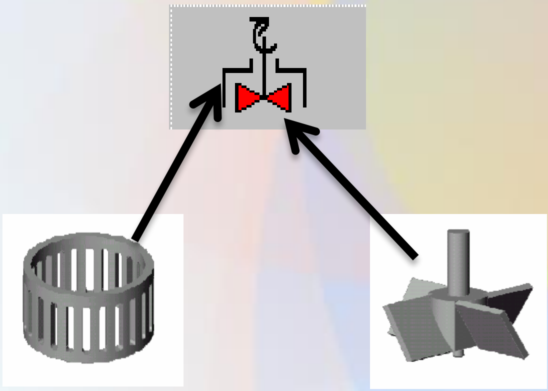
- **shear rates**
- **specific power**
- **pumping capacity**
- **power, etc.**

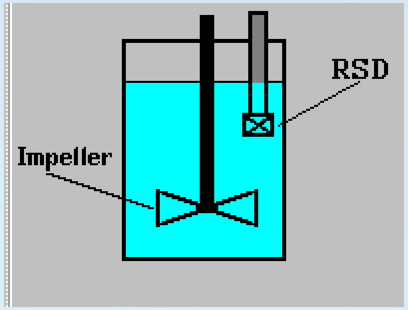
as functions of the equipment design and media properties for Newtonian and non-Newtonian liquid mixtures.

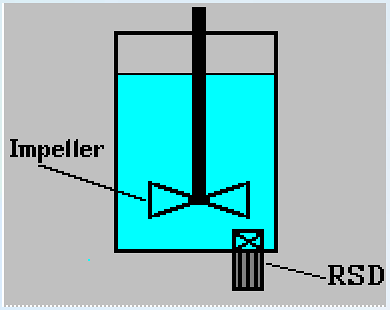
2) Modeling of batch and continuous flow homogenization of multicomponent media in mixing tanks and installations with agitators and RSD devices. Results of modeling include such parameters as

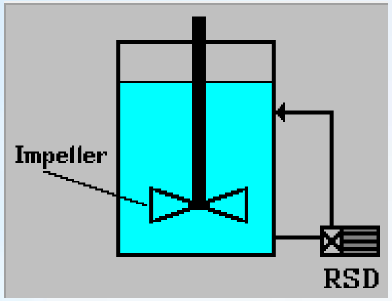
- **time required for different degrees of homogenization**
- **degree of homogenization achieved within the outlined process duration**
- **residence time distribution for media in high shear zone, etc.**

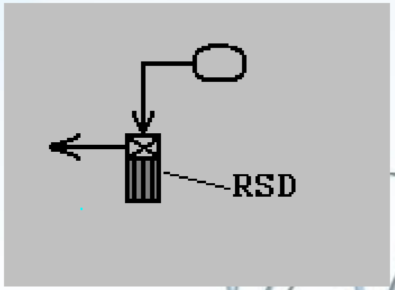
VisiMix RSDE











The **essence of the approach** consists in describing **the mean size of drops formed** in different flow conditions as a function of **the average values of turbulent energy dissipation rate** in the flow or vessel, estimated as specific power consumption per unit of mass of media:

$$\varepsilon_{av} = P / (\rho V)$$

A real progress could be achieved by analyzing the **kinetics of breaking and coalescence of droplets** with account to local hydrodynamic conditions.

Starting from 1996, this model is successfully used for practical application in the program VisiMix Turbulent {see www.visimix.com}.

Currently this model is used also as a base for mathematical modeling of breaking and coalescence of droplets in high shear channels of RSD devices.

KINETICS OF EMULSIFYING

At the current stage of the research:

- The task is limited to simplified modeling of kinetics of simultaneous breaking and coalescence of droplets in the range of diameters corresponding to Kolmogorov's (non-viscous) range of linear micro-scales of turbulence.
- The mixing was assumed to be "perfect", i.e. all positions of a drop in the tank were assumed to be equally probable, and distributions of drop sizes and concentration of the disperse phase were considered uniform.
- The system was assumed to be mono-disperse.

In such conditions, **the number of drops in a liquid-liquid system** with defined physical properties and constant volume fraction of the starting from some initial size, in a volume with non-uniform distribution of turbulence may be described by equation:

$$\frac{dd}{d\tau} = \frac{d}{3V}\int_V (N_c - N_b)dV$$

Three functions should be known:

- The two of them, **frequencies of coalescence and breaking**, N_c and N_b, depend on the drop size, physical properties of the phases and local rate of turbulent dissipation of energy.
- The third - **distribution of turbulent dissipation by volume** - depends on design and operational regime of the emulsifying device.

THE FREQUENCY OF BREAKING

An individual act of deformation and breaking must be assumed to occur under action of an instant **velocity pulsation in the vicinity of the drop** on the condition that the amplitude of the pulsation exceeds a certain minimum value v*.

The relation between this **"critical"** value and the mean square root velocity was estimated as

$$U^* = \frac{v^*}{\bar{v}} = \frac{0.775}{\varepsilon^{1/3} d^{1/3}} \left(M / d + \sqrt{\left[(M / d)^2 + \frac{10\sigma}{\rho_c d} \right]} \right)$$

$$M = \left| 1.2 \frac{\rho_d}{\rho_c} v_d - 3 v_c \right|$$

VisiMix
software for chemical engineering

The linear scale of the "destroying" pulsations was estimated as l = 2.17 d - **the minimum length of the deformed droplet**, corresponding to the loss of stability. Within the framework of this model, the mean frequency of drops breaking in an area with the local turbulent dissipation ε may be estimated as

N_b = *mean frequency of pulsations of the scale l*
x *relative frequency of pulsations l with amplitudes* v' ≥ v*
x *probability of one or more droplets residing in an area of the scale l*

$$N_b = f_l P(\mathrm{v}' \geq \mathrm{v}^*)(1 - P(0))$$

$$f_l = \frac{1}{l^3}\frac{\varepsilon^{1/3}}{l^{2/3}}$$

$$P(\mathrm{v}' \geq \mathrm{v}^*) \cong \sqrt{2/\pi}\int_{U^*}^{\infty} \exp(-U^2/2)dU$$

$$P(0) \cong 1 - \exp(-19.6\varphi)$$

VisiMix
software for chemical engineering

THE FREQUENCY OF COALESCENCE

The act of coalescence is usually assumed to occur if

(1)two droplets approach each other and collide and

(2)the "efficient“ collision, i.e. the amplitude of the fluctuation is high enough to overcome the resistance of a liquid film separating the drops:

$$N_c = \textit{frequency of collisions} \times \textit{efficiency of collisions}$$

The necessary condition of coalescence of two droplets may thus be assumed to consist in their being **in contact as the fluctuation occurs.** The term "in contact" here means that the distance between the drops’ centers is practically equal to the drop diameter, d, and their surfaces are separated by a thin layer of ions existing on the water-oil boundary, water side.

VisiMix software for chemical engineering

According to the postulates of the DLFO-theory, **the interfacial boundary is surrounded with a "double layer" of ionized liquid**. Due to inter-action of these layers, the neighboring surfaces are kept from junction by electrostatic repulsive pressure, p. **The value of this pressure depends on the chemical composition of substances**. The coalescence only happens if the squeezing pulsation pressure is high enough to overcome the repulsive pressure.

The condition for a random turbulent pulsation to be **"efficient“** may thus be formulated as

$$v'_n \geq v_c{}^* = \sqrt{(2p \ / \rho_c)}$$

where v'_n is the constituent of the pulsation velocity v'_λ, normal to the contact surface, and $\lambda = d$ is the linear scale of the "coalescing" pulsations.

VisiMix
software for chemical engineering

According to this model, mean frequency of coalescence may be defined as

Nc = *mean frequency of pulsations of the scale* λ

x *relative frequency of pulsations with amplitudes satisfying the condition* $v'_n \geq v_c^*$

x *probability of the presence of two or more drops in an area of the scale* λ

$$N_c = f_\lambda P(\mathrm{v_n}' \geq \mathrm{v}^*)(1 - P_\lambda(0) - P_\lambda(1))$$

$$f_\lambda = \frac{1}{\lambda^3}\frac{\varepsilon^{1/3}}{\lambda^{2/3}}$$

$$P(\mathrm{v_n}' \geq \mathrm{v_c}^*) = \frac{1}{\sqrt{2\pi}}\int_{V^*}^{\infty}(1 - \frac{V'}{V^*})\exp(-V'^2/2)dV'$$

$$V' = \mathrm{v}'_\lambda / \bar{\mathrm{v}}_\lambda$$

$$V^* = \frac{\mathrm{v}_c^*}{\bar{\mathrm{v}}} = \frac{\sqrt{(2p/\rho_c)}}{\varepsilon^{1/3}\lambda^{1/3}}$$

$$1 - P_\lambda(0) - P_\lambda(1) \cong 1 - (1 + \varphi)\exp(-\varphi)$$

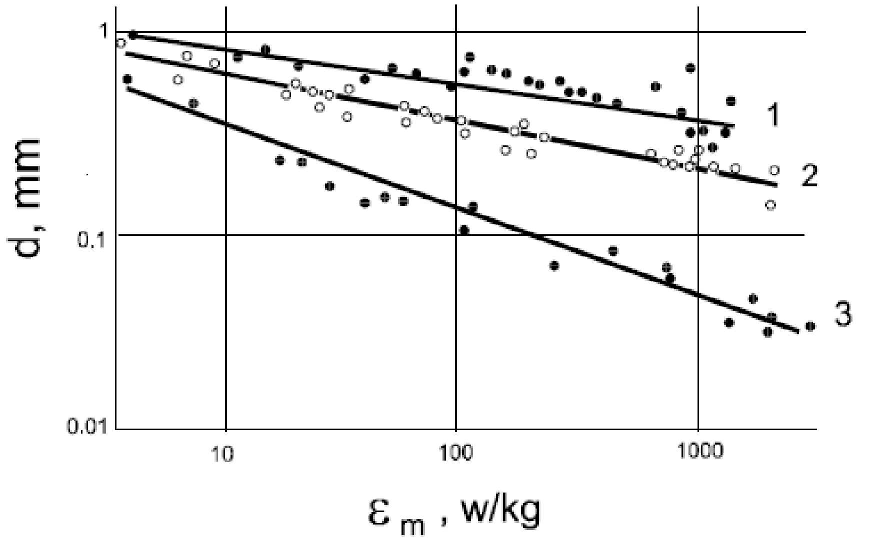


Figure 1. Mean drop diameter vs. ε_m. 1 - P = 20 Pa; 2 - P = 7 Pa; 3 - P $\rightarrow \infty$ (fully stabilized). Solid lines correspond to calculations by equations 1-14.

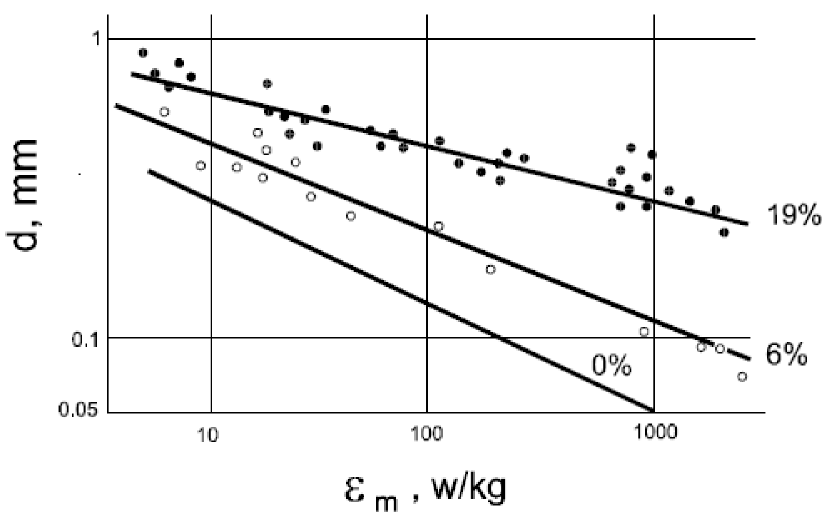


Figure 2. Mean drop diameter vs. ε_m. The effect of the concentration of the disperse phase. Solid lines correspond to calculations by equations 1-14.

Aplication

- Example 1: ***Batch emulsifying in a vessel with bottom-entering rotor/stator homogenizer***
- Example 2***: Emulsifying with in-line RSD. Scaling-up.***

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

software for chemical engineering

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

What do we do?

- 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

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

Simulant Testing

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

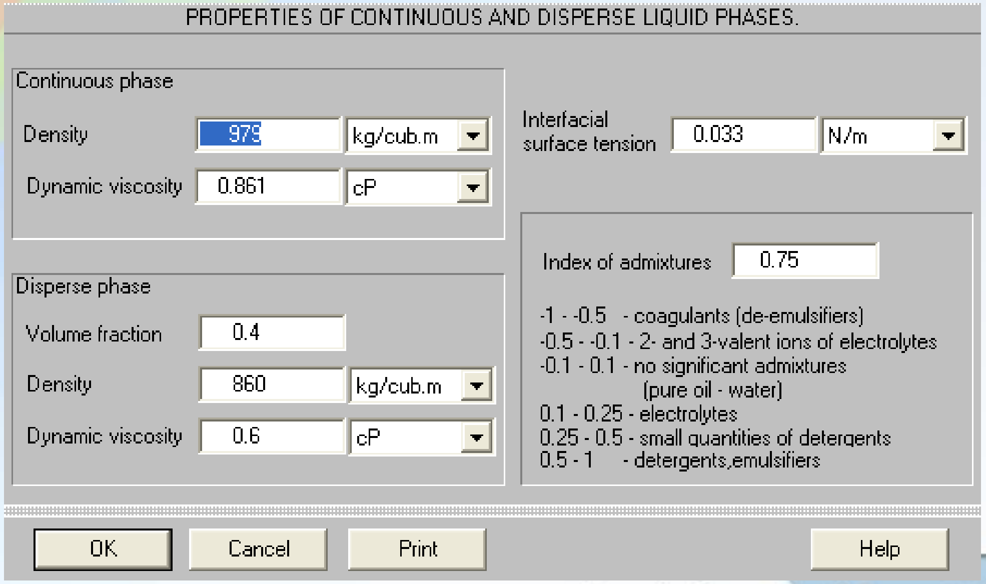
Existing Setup
"Disperserator"

Proposed Setup
Traditional Impellers

VisiMix
software for chemical engineering

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.



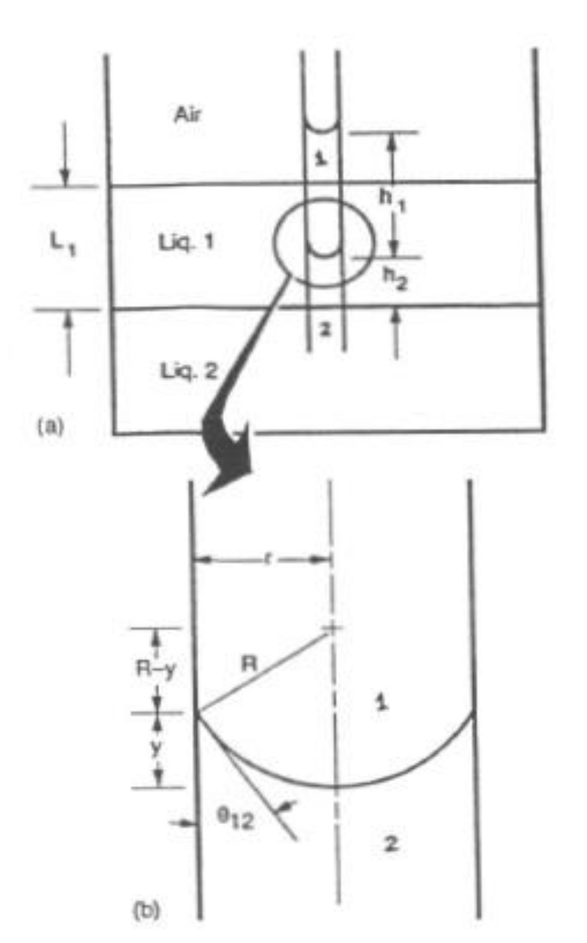
Required Inputs

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



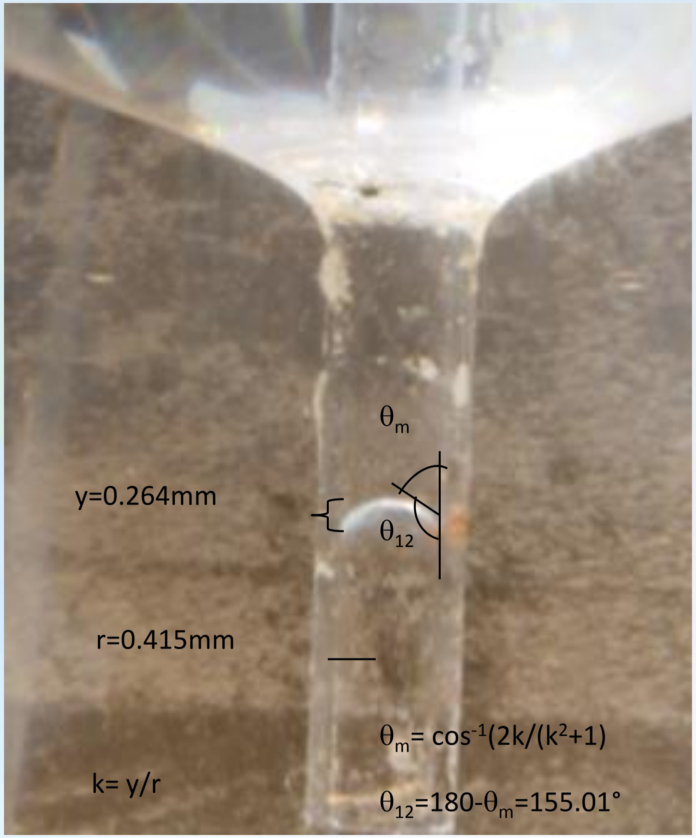
Required Inputs

- 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

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

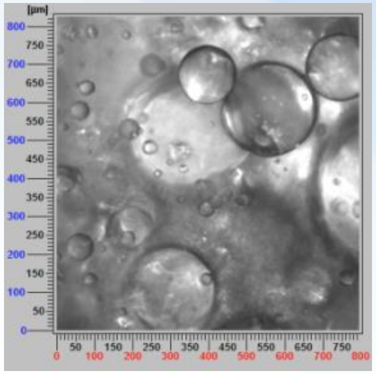
- Photograph of Toluene/water interface
- Measured interfacial tension our system (Toluene/Water)
  - 0.0327 $N{\cdot}m^{-1}$
- Reported/reference interfacial tension for Toluene/Water
  - 0.0364 $N{\cdot}m^{-1}$.

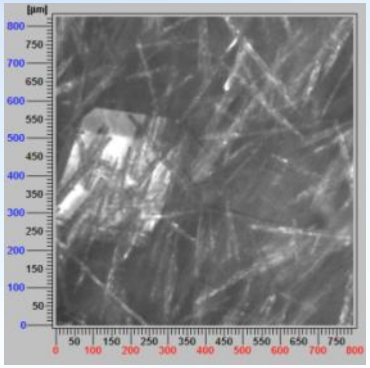


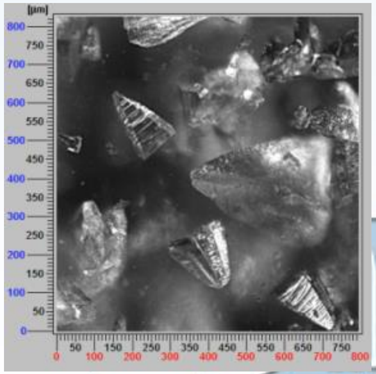
Particle Vision Microscopy: **PVM**

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

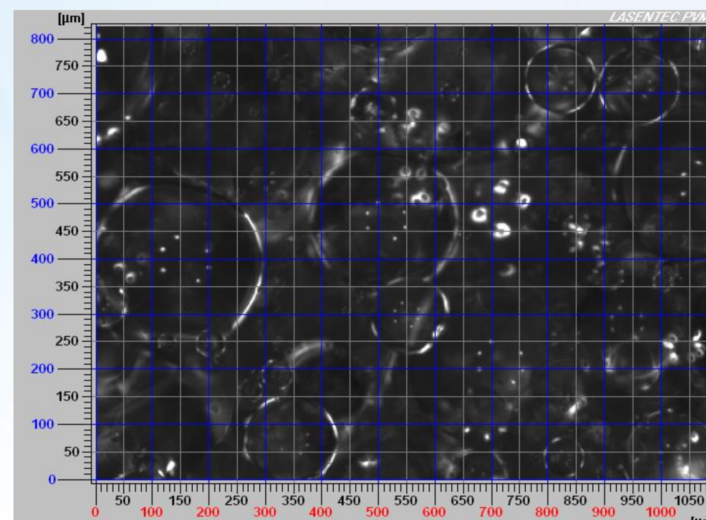






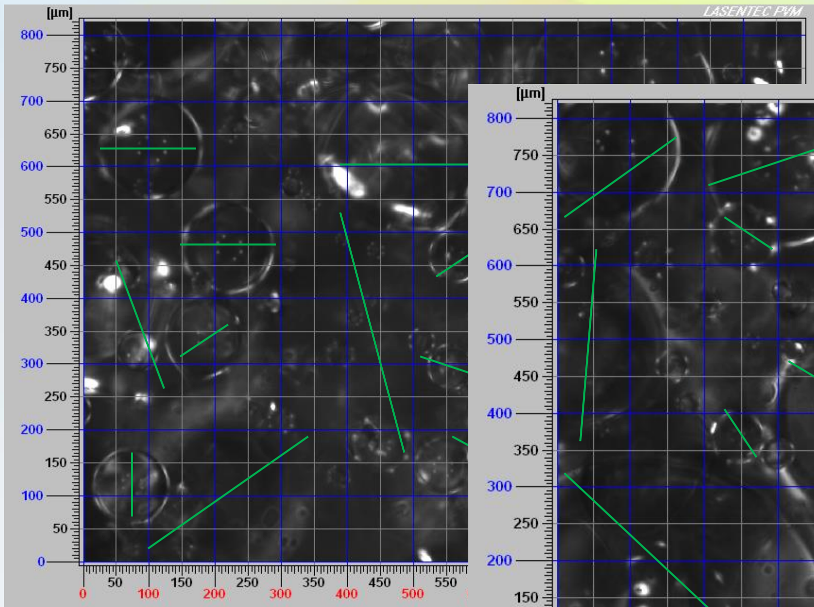
Validate Model Using PVM

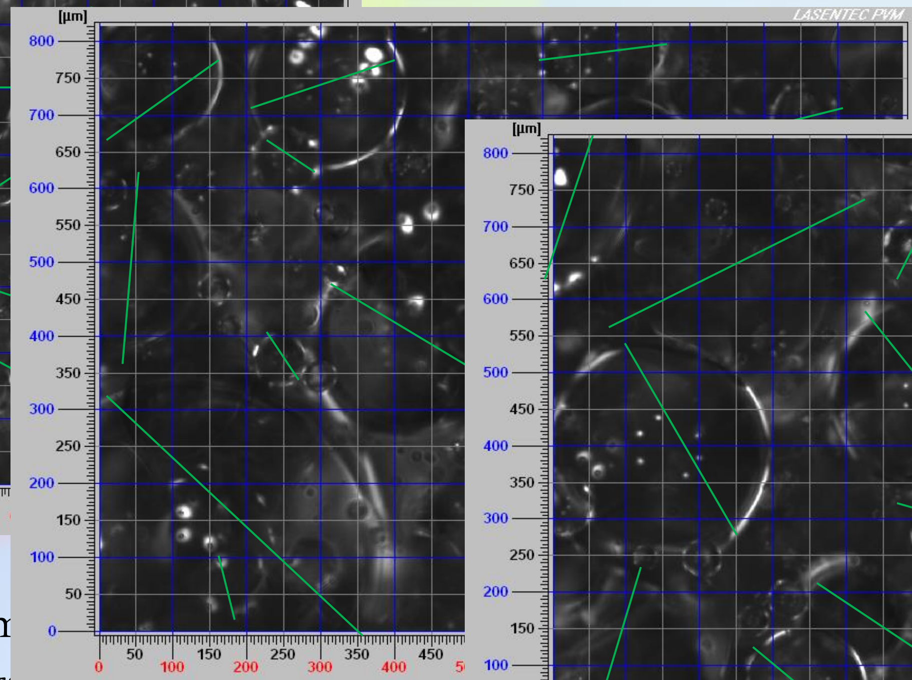
- Taking the PVM data at one setup to test the model for the admixture value.
- Comparing drop size distribution to the VisiMix values
- By matching the shear between systems we hope to match drop size, surface area, and mixing.
  - Mean drop size

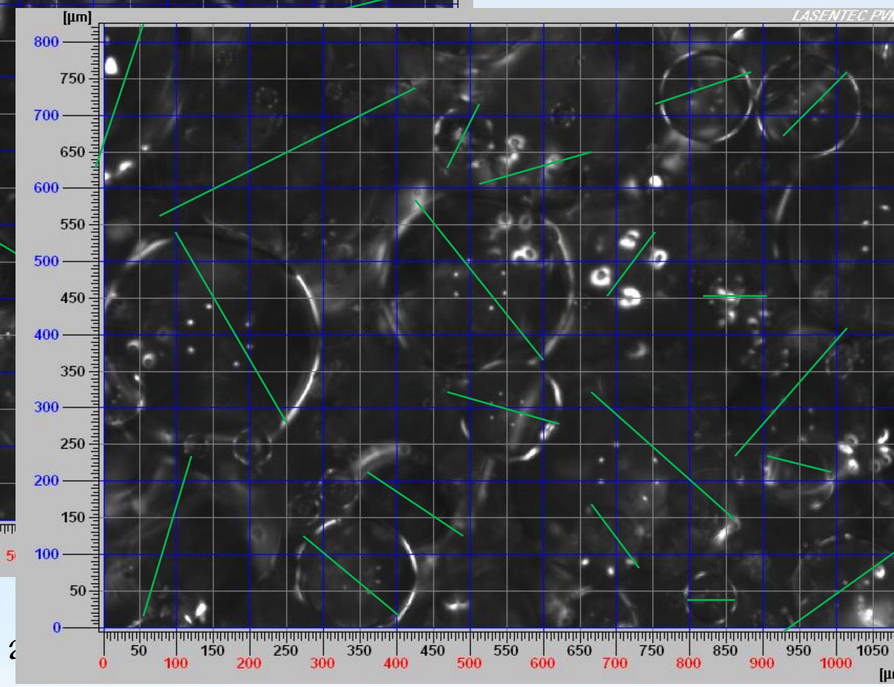


software for chemical engineering

Calculating Drop Diameter from PVM







Average of drop diam
PVM image

Repeat with a

Repeat a third time.

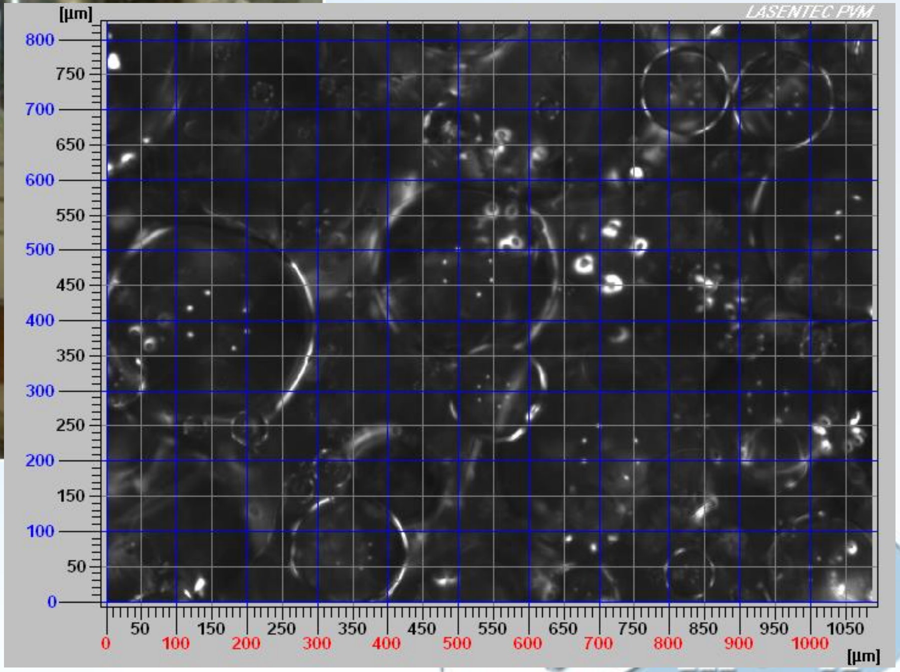
The average diameter for all three images is then averaged again and that value is the drop diameter for that RPM

VisiMix
software for chemical engineering

RC-1 Experiments

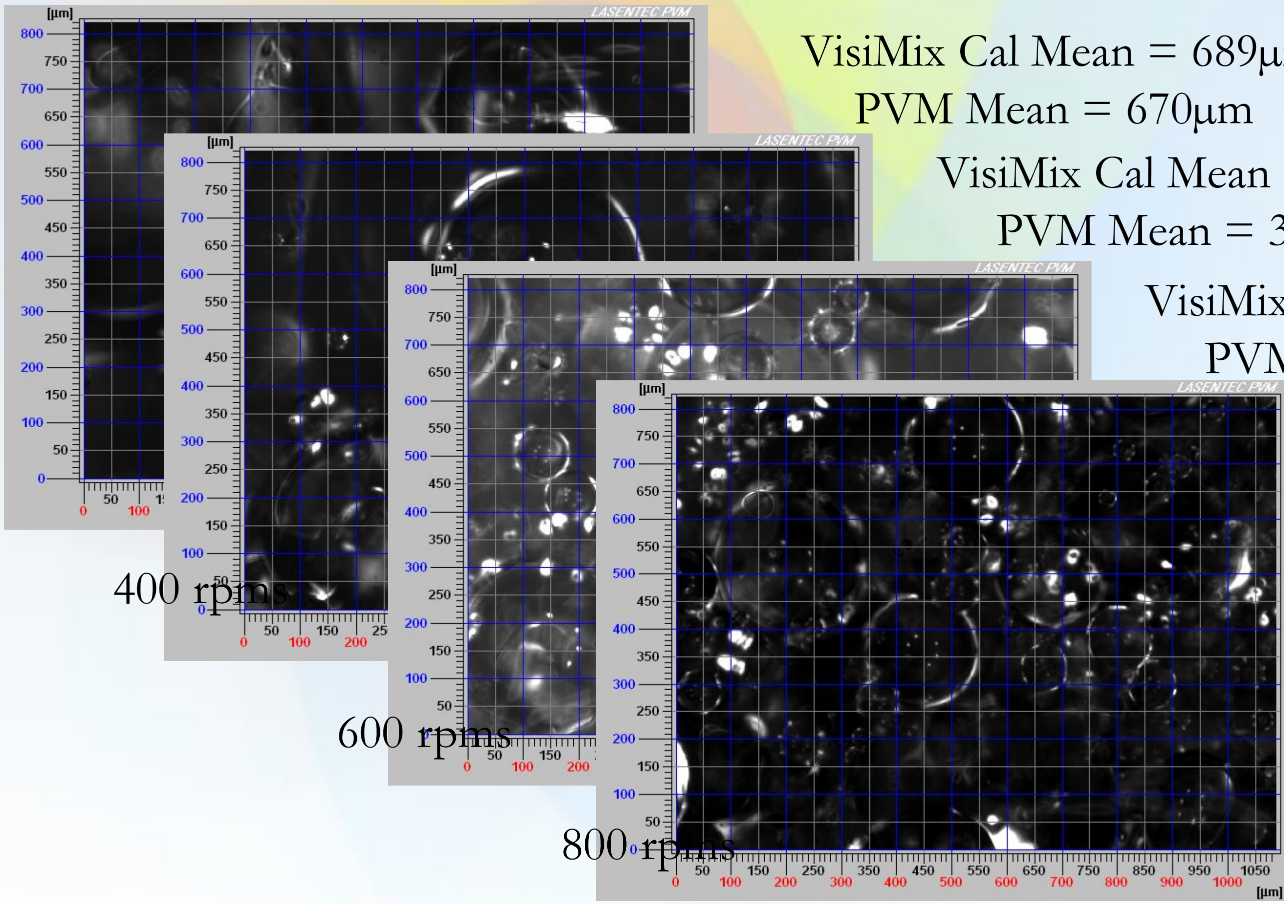
Pitch blade impeller with PVM and Tr as baffles.

PVM mean ≈ 280 μm



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

RC-1 Experiments Using PB-Impeller



400 rpms

600 rpms

800 rpms

10000 rpms

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

VisiMix
software for chemical engineering

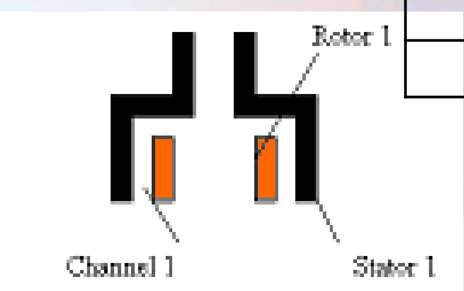
VisiMix RSD

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

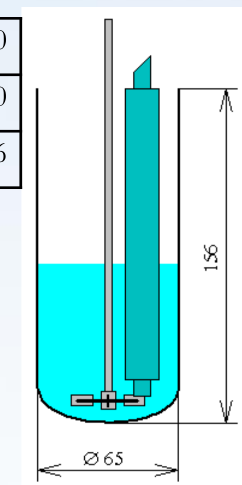
Modeling

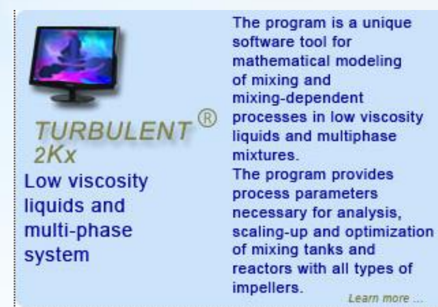
- VisiMix models both traditional type impellers (Turbulent 2K) and rotor stator mixers (RSD)
- First calculate mixing parameters using rotor stator model
- Match the output using Turbulent 2K
  - Trial and error by simply changing rpm



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

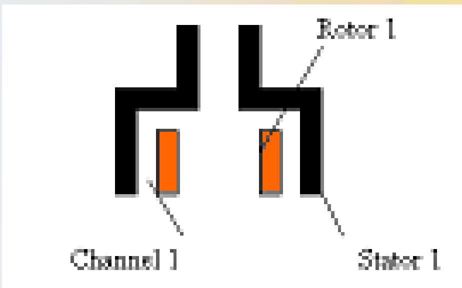


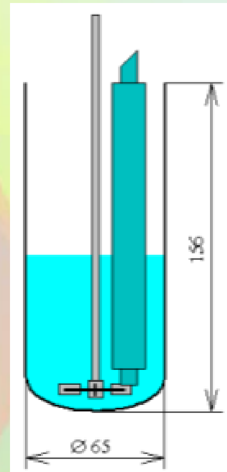


VisiMix
software for chemical engineering

Disperserator Experiments

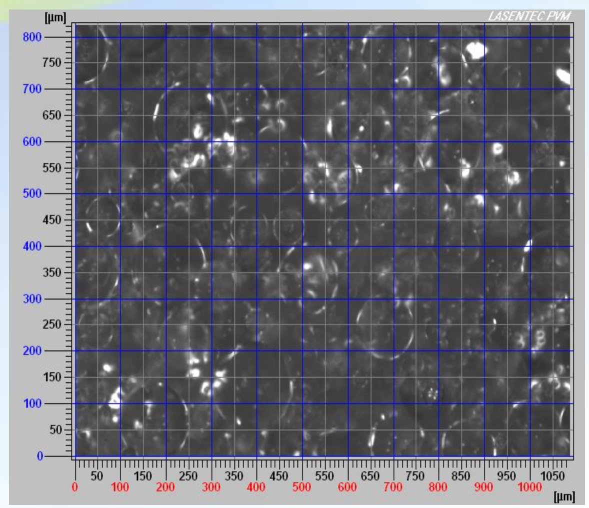
Modeling:





Mean diameter
=138 µm

Experimentation:



Mean diameter
≈ 130 µm

VisiMix
software for chemical engineering

Conclusions

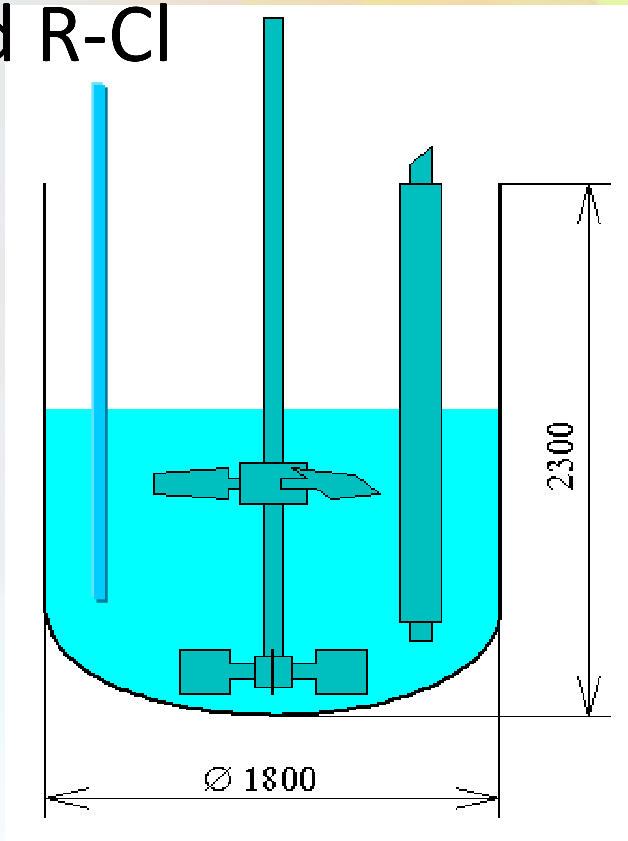
- **VisiMix** accurately predicts mixing parameters for both traditional impellers and **rotor/stator systems for liquid-liquid mixing**
- By modeling the dispersion in the historical laboratory equipment we are able to identify automated reactor configurations that will maintain the same degree of mixing.

High Shear Rate at Chemical Fast Reactions

Process and Quality Problem

R-6826

Feed R-Cl



Process

$R\text{-}NH_2 + R'\text{-}Cl \longrightarrow t\text{-}D\text{-}R\text{-}R'$

Impurity

t-L-R-R’

Impurity results at laboratory and in production

[%]impurity	RPM	Impeller type	volume	System
0%	15,000 rpm	rotor stator	0.63 lit	Laboratory reactor
0.3%	1,500 rpm	3-blade		
0.6%	800 rpm			
1.5%	100 rpm			
0.3% - 0.6%	140 rpm	bottom – flat blade up - turbofoil	2,978 lit	Production R-6826

Correlation between shear rates and the impurity concentration

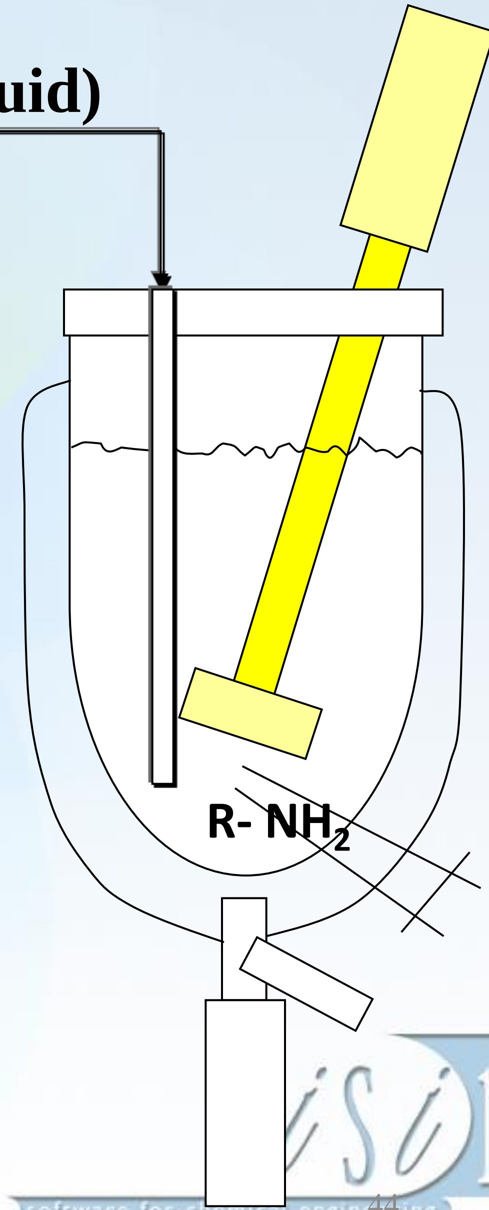
Working with rotor stator at laboratory scale

Problem

How to scale up ?

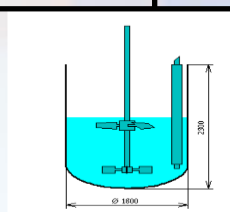
Potential Saving :

MORE than 250 K$

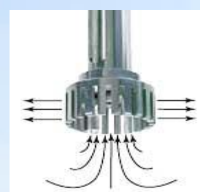


Calculating shear forces with VisiMix

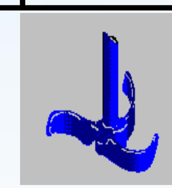
Turbulent shear rate [1/s]	[%]impurity	RPM	Impeller type	system
780,000	0%	15,000 rpm	rotor stator	Laboratory reactor
32,900	0.3%	1,500 rpm	3-blade	
12,900	0.6%	800 rpm		
580	1.5%	100 rpm		
15,200	0.3% - 0.6%	140 rpm	bottom – flat blade up - turbofoil	R-6826



R-6826



Rotor stator



Lab impeller

The required shear rate can not be achieved in the production reactor

VisiMix Products

The latest VisiMix products are:

- **VisiMix 2K8 Turbulent**
- **VisiMix 2K8 Laminar**
- **VisiMix 2K8 Different Impellers**
- **VisiXcel- Data Base**
- **Pipe Line**
- **Rotor Stator Disperser – RSD**

NEW!

VisiMix Orientation

VisiMix Demonstration Tools

The VisiMix Demonstration Tools:

- ✓ VisiMix Turbulent – Examples & User Guide
- ✓ VisiMix Laminar - Examples & User Guide
- ✓ VisiMix Different Impellers – Examples & User Guide
- ✓ VisiMix RSD– Examples & User Guide
- ✓ VisiMix Turbulent SV – Trial & Education
- ✓ VisiMix Review of Mathematical Models
- ✓ Selected Verification Examples

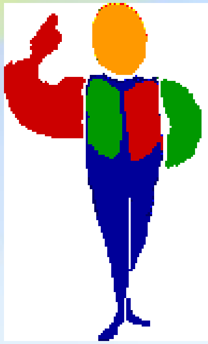
  The Comparison between Published Experimental Data and VisiMix Calculations

http://www.visimix.com

Conclusion

- Using VisiMix Products support you can
  - understand better your processes
  - Reduce dramatically your Scaling up processes and Scaling down
  - Save a huge amount of Time & Money ($1,000,000 +)
- The VisiMix Products are friendly and easy to use with very quick results.
- The VisiMix results are based on a systematic and seriously experimental checking – and found very reliable.
- VisiMix Projects Parameters and Data Base allows you to share and transfer the data with colleagues in the company.

Thank you for your attention



VisiMix
software for chemical engineering