

# 2011 VisiMix International Conference: The Influence of Mixing in Your Process

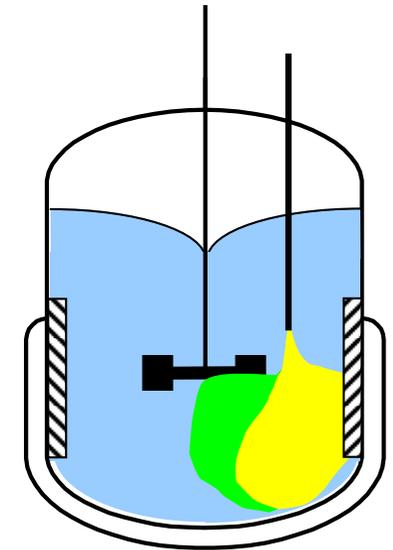
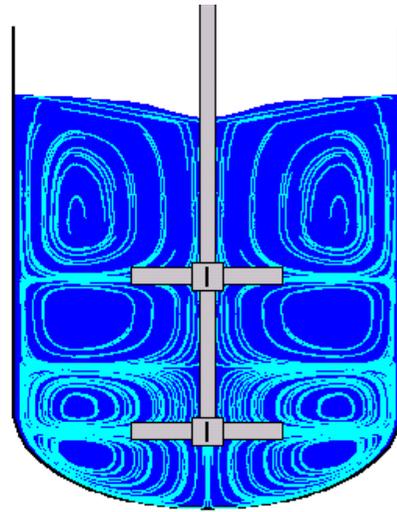
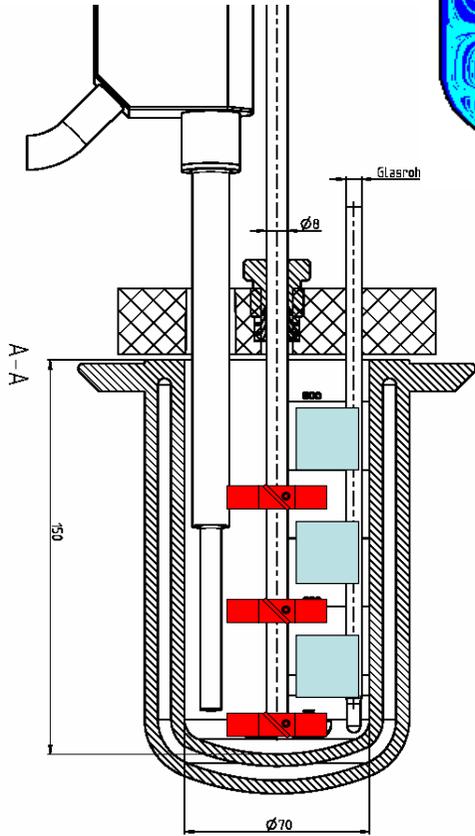
Boston USA

July 13-15, 2011

rm<sup>2</sup>technologies LLC

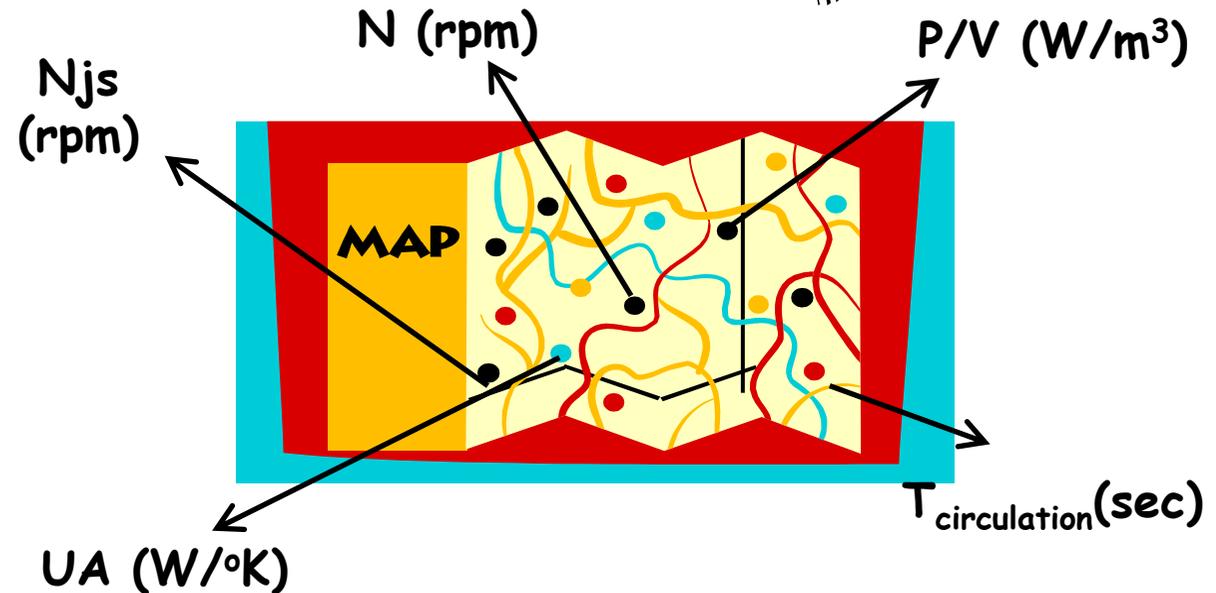
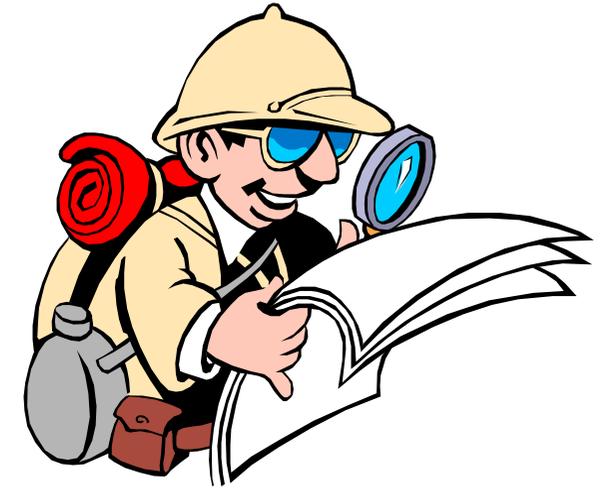
Application of Visimix to the Characterization of Lab Reactors

Reinaldo M. Machado



# Some questions we would like to answer before we scale-up...

- What was going on in our lab reactor?
  - *Where have we been (Lab)?*
  - *Where are we going (Plant)?*
- How do we make the lab and plant reactor perform the same?
- What controls the rate of our process?
  - Kinetics
    - Rate of addition
    - temperature
  - Mass-Transfer
    - Gas/Liquid
    - Solid/Liquid
    - Liquid/Liquid
  - Blending
  - Heat Transfer



## Agitation intensity and agitator equipment design impacts many fundamental rate processes

- **Blending and macro-mixing**
  - Impeller pumping flow rate
- **Mass transfer**
  - Power/volume
  - Impeller pumping flow rate
- **Heat transfer**
  - Impeller velocity
- **Kinetics & selectivity (fast reactions)**
  - Local concentration gradients from semi-batch feed tube
  - Impeller pumping flow rate
  - Power/volume
- **Phase dispersion (solid/liquid, gas/liquid, liquid/liquid)**
  - Power/volume
  - Kinetic energy
  - Shear rate

**Scale-up is about balancing different process rates as they change with process size**

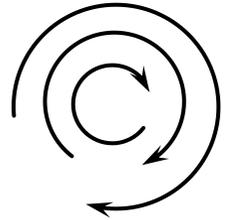
**Unfortunately the process rates do not change uniformly with volume and equipment size! It is best to scale-up with one key mixing variable.**

# Generally three mixing length and time scales are used to describe the scale-up and scale-down of single phase stirred reactor processes in turbulent flow

- **micro-mixing**

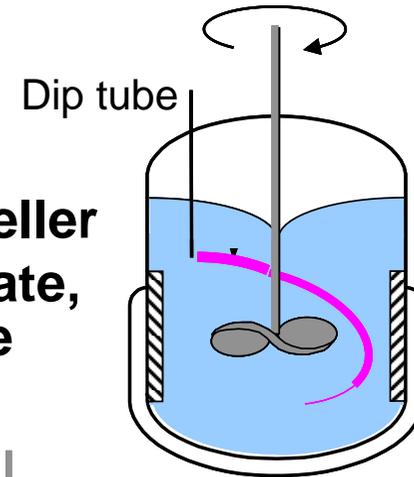
- at the scale of turbulence
- function of viscosity, density and power/volume

Turbulent Eddy diameter =  $f(P/V)$   
 micron scale; interior = laminar Flow



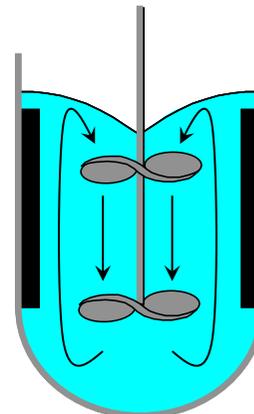
- **meso-mixing**

- at the scale of the feed tube and impeller
- function of feed tube location, feed rate, power/volume, impeller pumping rate



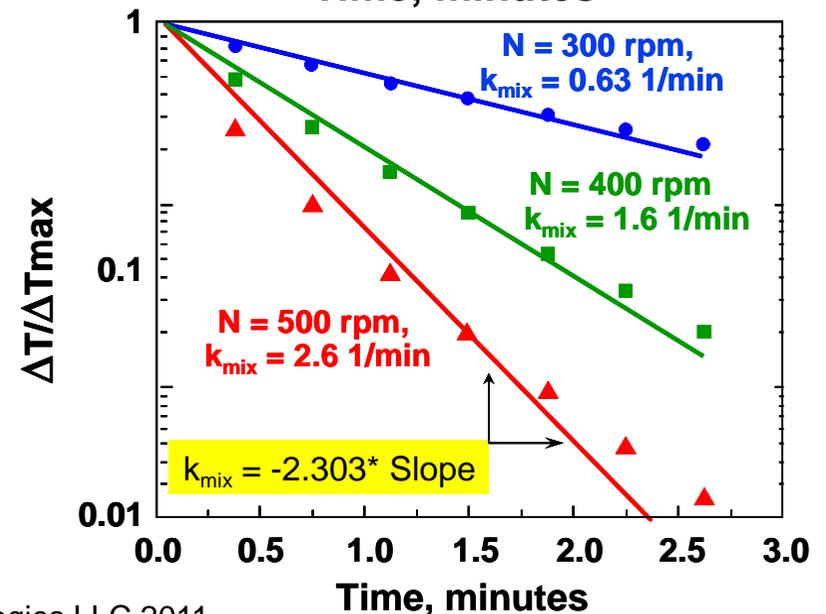
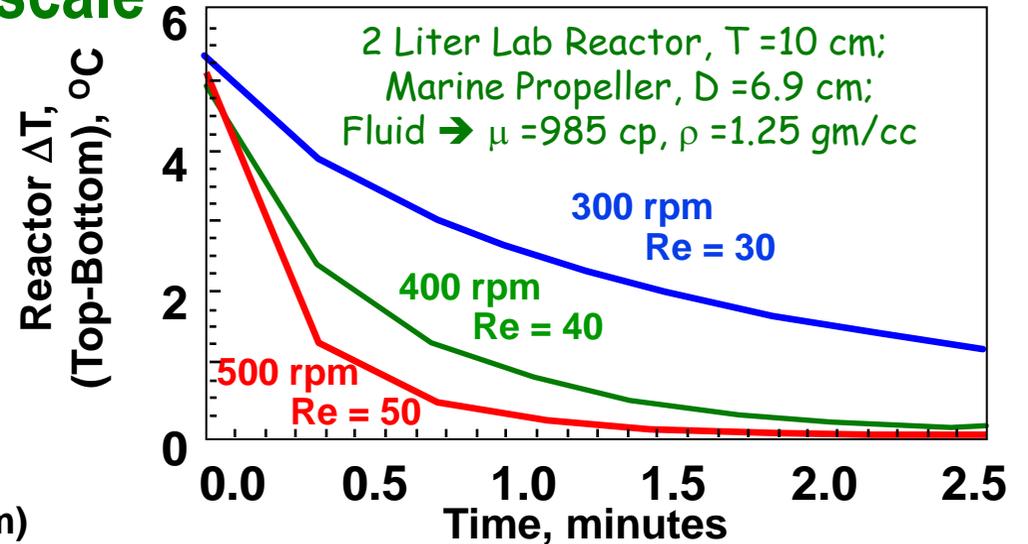
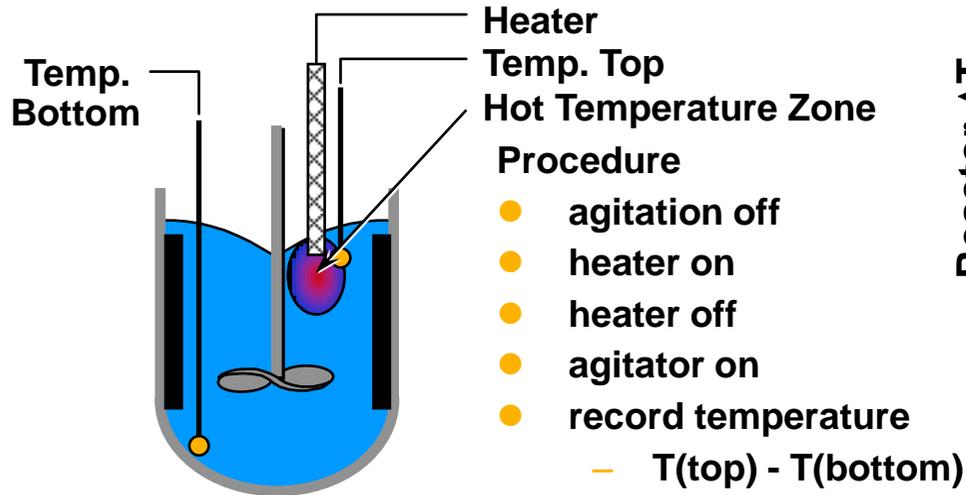
- **macro-mixing**

- at the scale of the reactor
- function of feed rate, impeller pumping rate



# Blending macro-mixing

Macro-mixing describes the uniformity of composition and temperature on the reactor scale



$\Theta_{mix}$  = Time to achieve a desired uniformity

N = Agitation rate

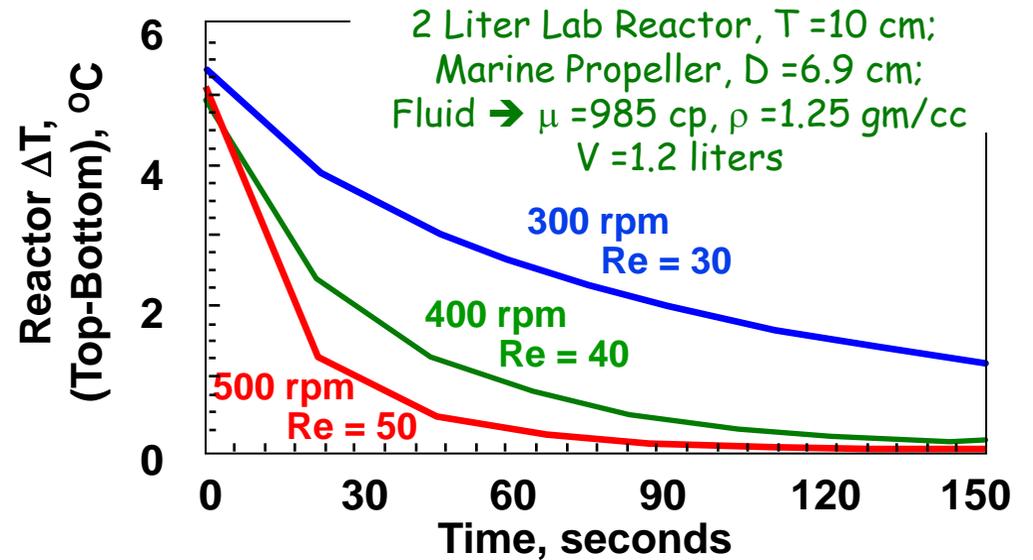
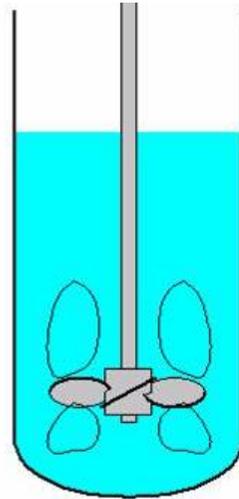
$$\Theta_{mix} \propto \frac{1}{N}$$

Mixing can be correlated with

$$N \bullet \Theta_{mix}$$

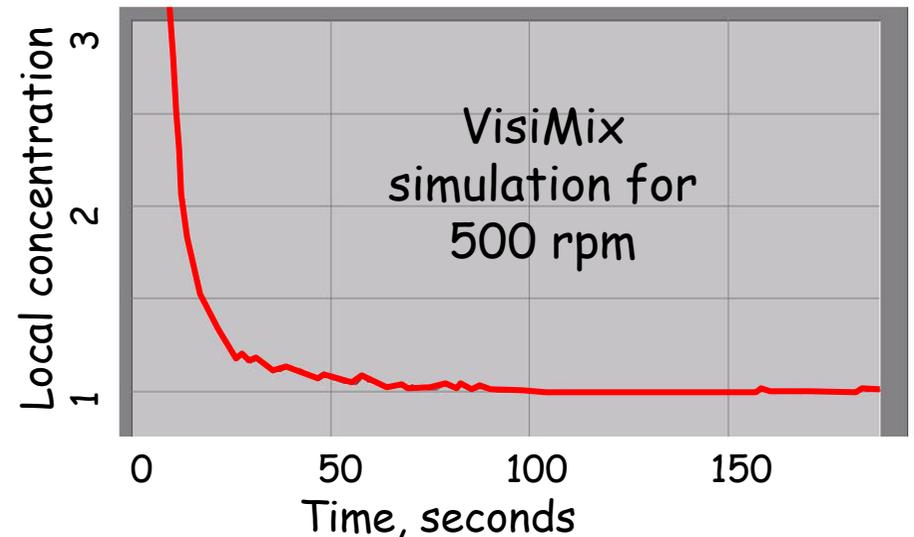
# Estimating blend time in the Mettler-Toledo RC1 MP10 reactor

VisiMix model of MP10 showing "mean" velocity pattern at 500 rpm



	Q l/min	uniformity index 0.2 to 1	time, seconds, to achieve 90% uniformity	
rpm	VisiMix		lab	VisiMix
300	4.2	0.29	220	146
400	5.7	0.31	86	92
500	7.4	0.34	53	64

Characteristic function of tracer distribution



# Alternate thermal method for mixing characterization of lab reactors: Rapid injection of cold feed during agitation

½ life to reach final conditions

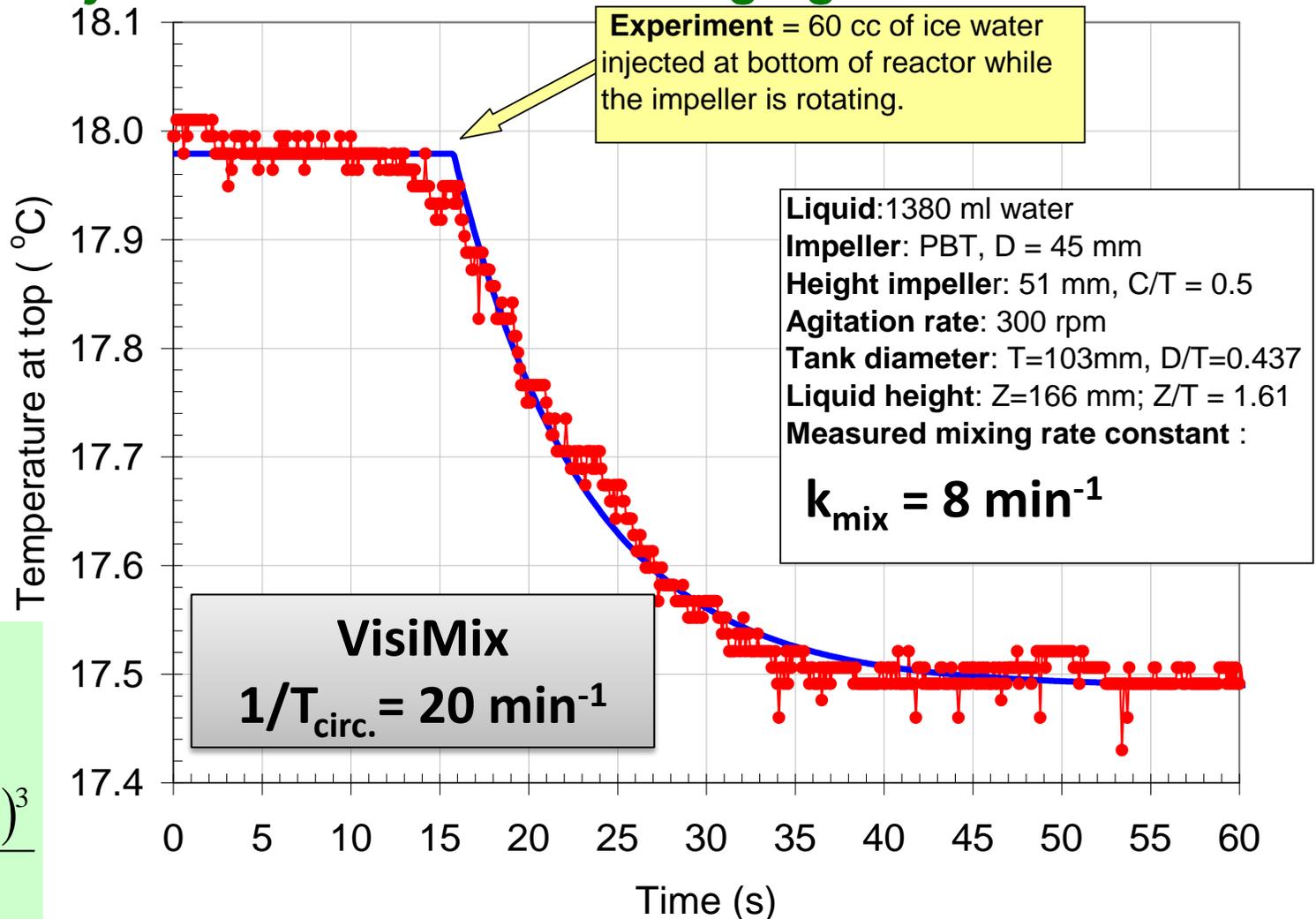
Experiment (Thermal) = 5 seconds  
 VisiMix (Composition) = 2 seconds

Estimate based on impeller pumping

$$k_{mix} = \frac{Q_p}{V_{reactor}} = \frac{N_Q ND^3}{V_{reactor}}$$

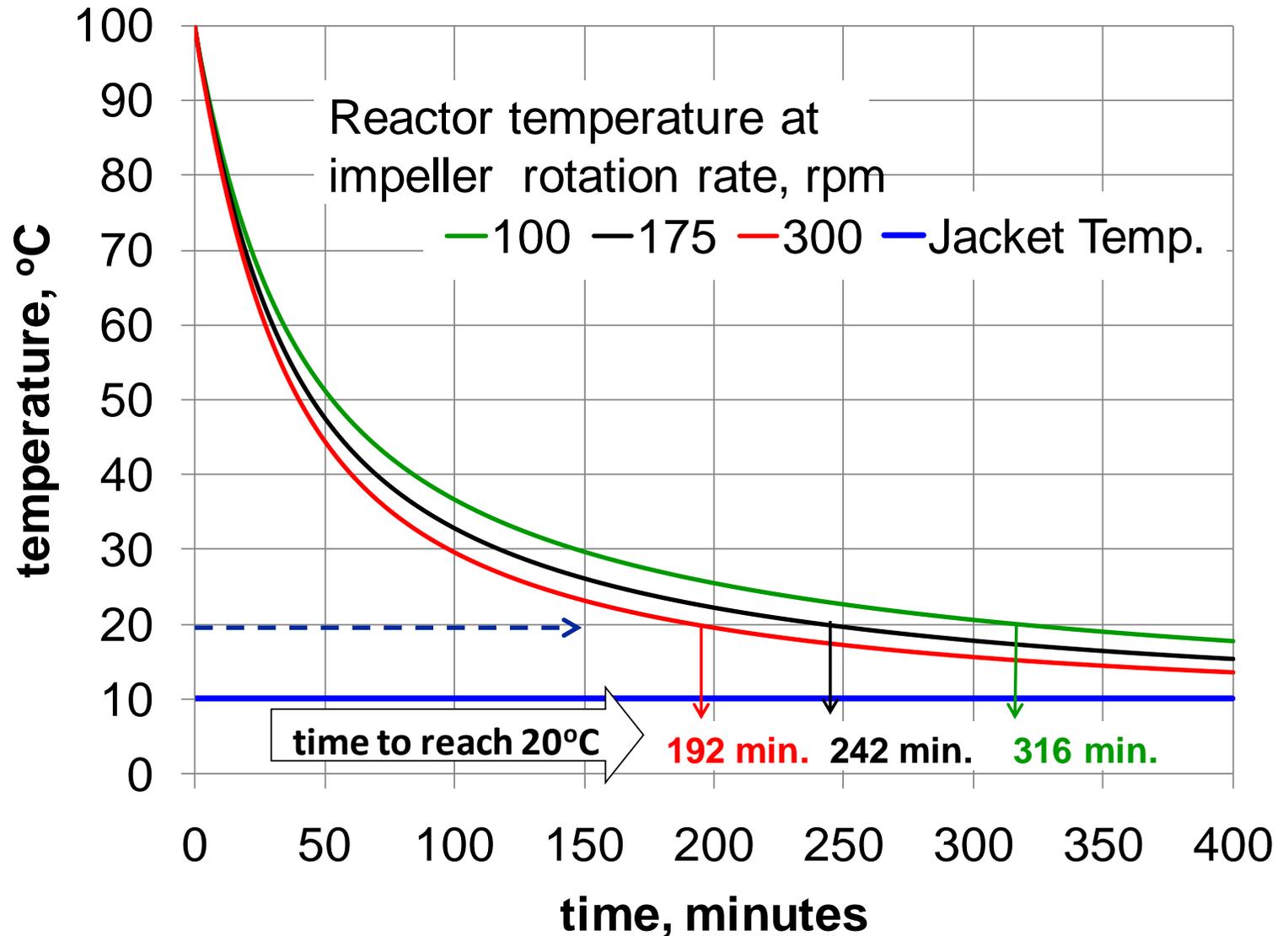
$$k_{mix} = \frac{0.6 \cdot 300rpm \cdot (4.5\text{ cm})^3}{1440\text{ cm}^3}$$

$$k_{mix} = 11\text{ min}^{-1}$$



## Cooling curves for glycerol in a stirred tank reactor

How long will it take to cool the reactor contents to 20°C?



# Heat Transfer Coefficient for glycerol in stirred tank

$$Q_{removal} = UA \cdot (T_{reactor} - T_{jacket(average)})$$

where,

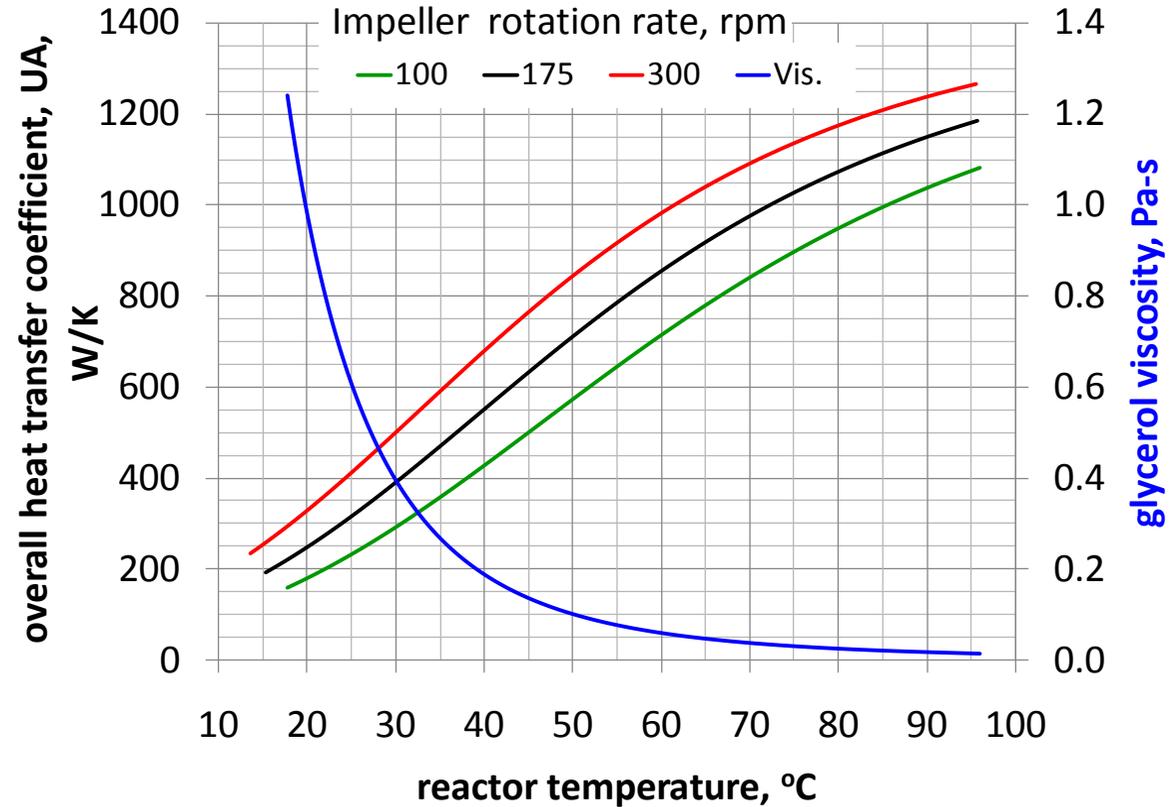
$Q_{removal}$  = heat removed by the cooling fluid, (W)

$U$  = heat transfer coefficient, ( $W/m^2K$ )

$A$  = heat transfer area contacted by the reaction mass, ( $m^2$ )

$T_{reactor}$  = temp. of the reaction mass, (K)

$T_{jacket(average)}$  = average temp. of the coolant in the reactor jacket, (K)



**The heat transfer coefficient decreases as the reactor cools and the glycol becomes more viscous.**

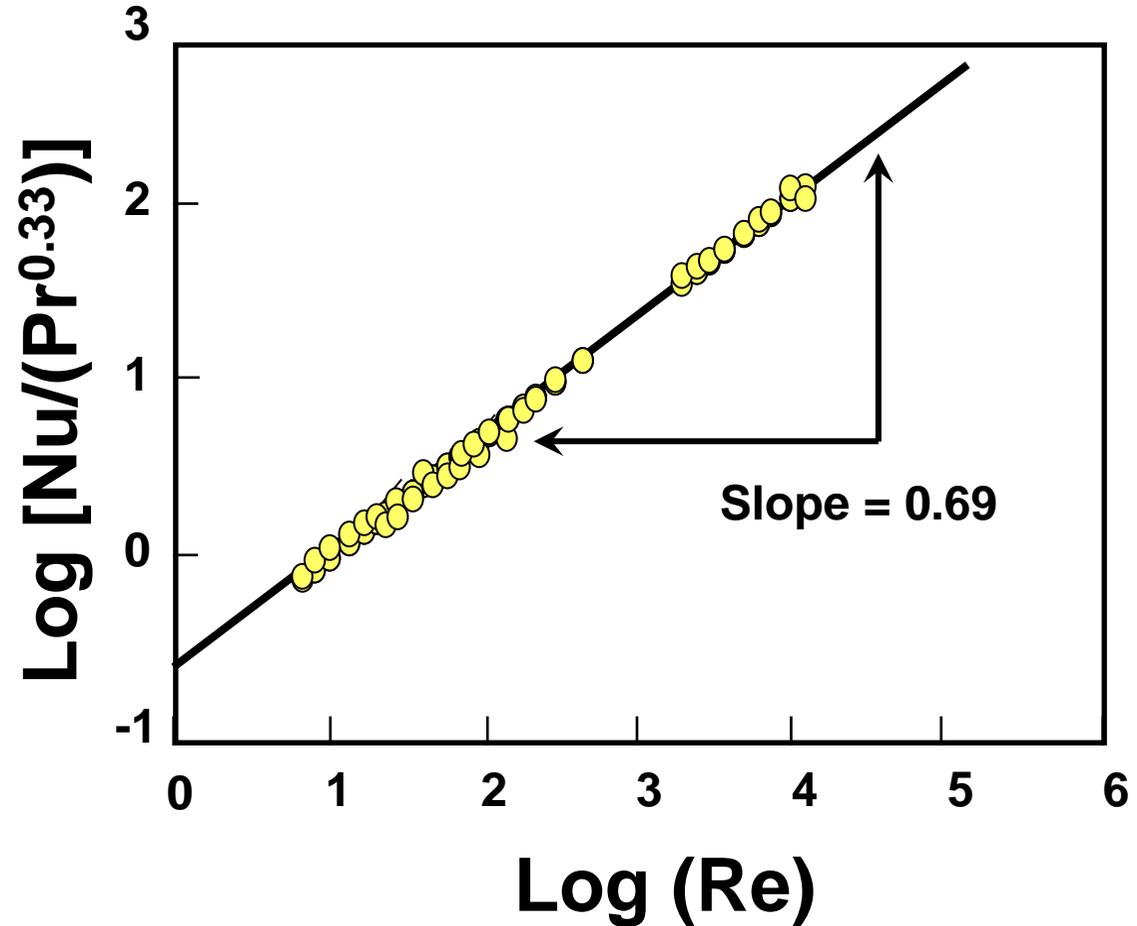
# Heat transfer characteristics of RC1 MP10 is similar to large scale reactors

$$Nu = C \cdot Pr^{1/3} \cdot Re^{2/3} \cdot \left( \frac{\mu_{fluid}}{\mu_{wall}} \right)^{0.14}$$

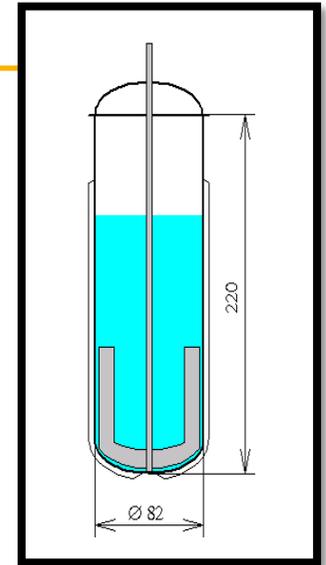
$$Pr = \frac{\mu_{fluid} C_p_{fluid}}{k_{fluid}} = \text{Prandtl Number}$$

$$Re = \frac{\rho_{fluid} N D^2}{\mu_{fluid}} = \text{Reynolds Number}$$

$$Nu = \frac{h_{fluid} T_{Tank Diameter}}{k_{fluid}} = \text{Nusselt Number}$$



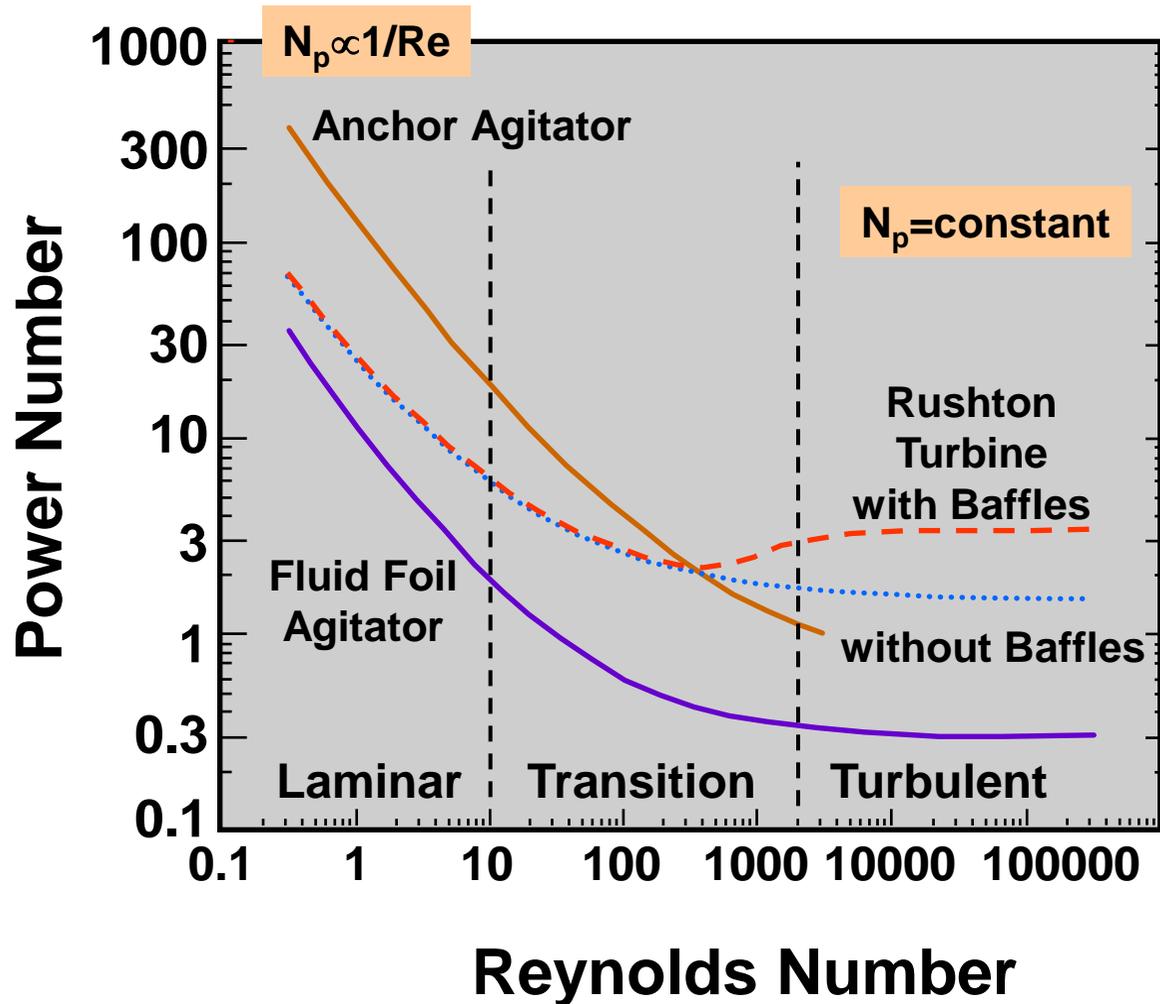
# VisiMix predictions and Experiments for Heat Transfer with Anchor Impeller with Glycerol in the RC1 MP10 glass lab reactor



	Inlet Jacket Temp. °C	Reactor Temp. °C	Reactor fluid $\mu$ , cP	Re	Overall Heat transfer coeff. U W/(m <sup>2</sup> K)	Heat removal rate, W
Experiment	14	25	940	13	58.1	25.0
VisiMix					59.5	27.0
Experiment	31	40	274	43	68.4	25.0
VisiMix					70.5	26.2
Experiment	48	55	89	133	83.7	25.0
VisiMix					80.0	23.2

# Mechanical power input

One way to characterize agitation intensity uses universal power curves to describe impeller performance



$$Re = \frac{\rho N D^2}{\mu}$$

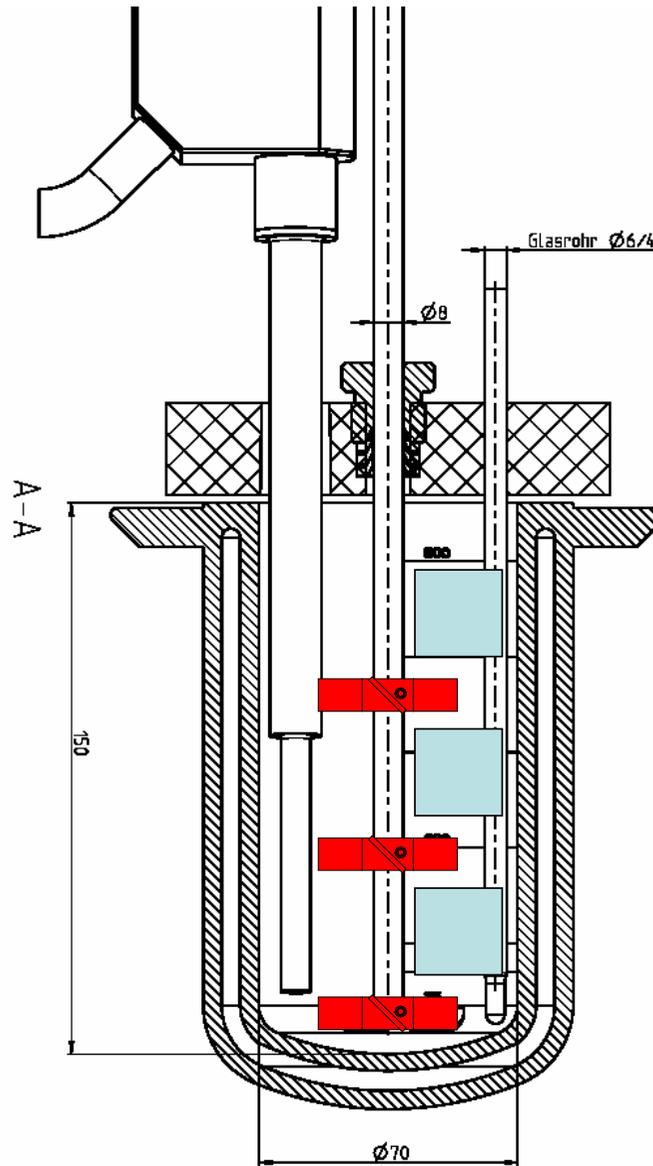
$$= \frac{\text{Inertial Force}}{\text{Viscous Force}}$$

$$N_P = \frac{P}{\rho N^3 D^5}$$

Lab reactors may operate in Transition and Laminar flow while the plant operates in Turbulent flow!

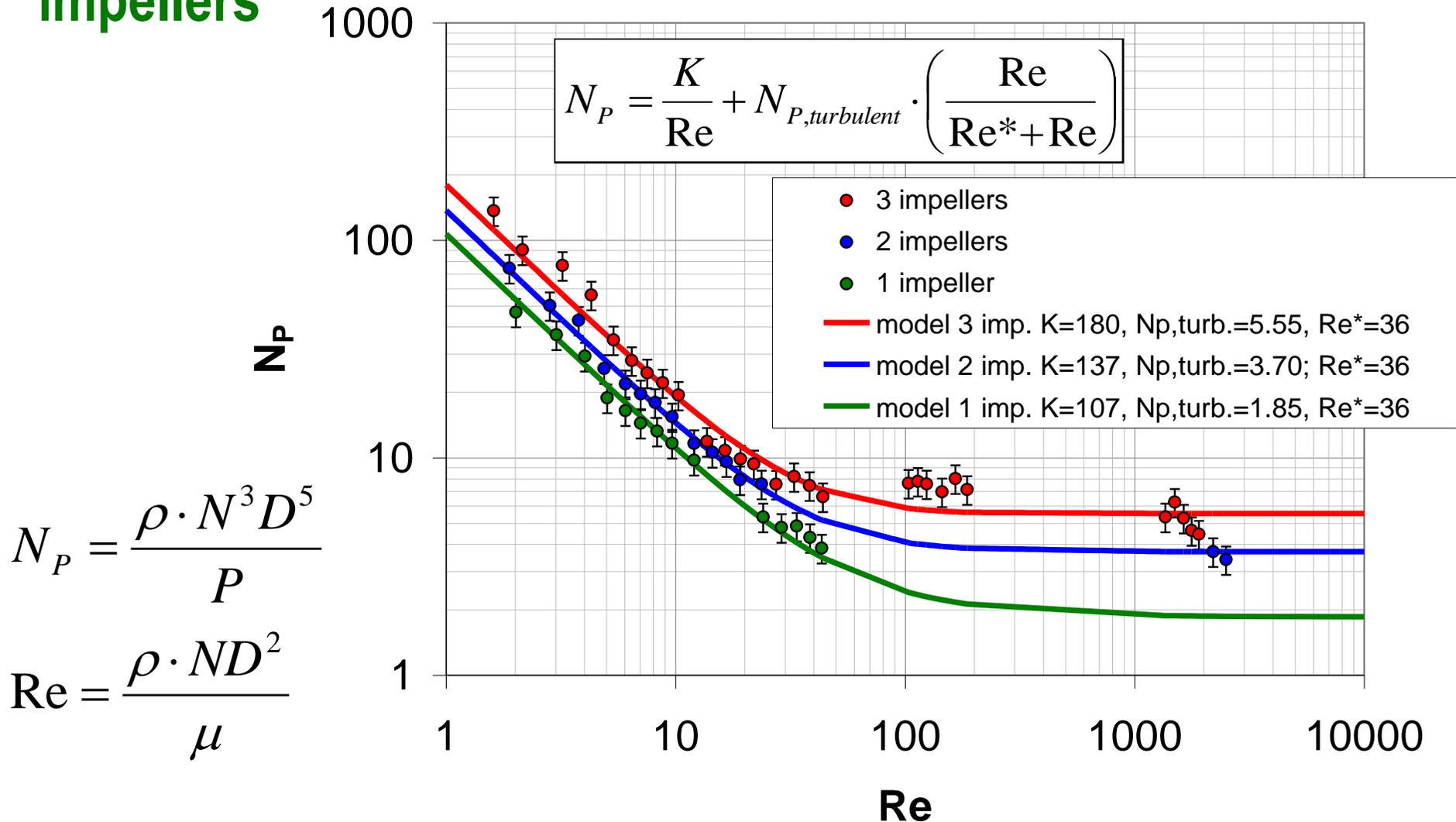
# Mettler Toledo AP01-0.5

The unique baffle and agitator design is appropriate for mixing diverse liquid chemical systems, from high-viscosity laminar flow to low-viscosity multi-phase turbulent flow.



- **Reactor**
  - T = 70 mm
  - Maximum recommended liquid height Z=140 mm
- **Impeller**
  - 1, 2 or 3 depending up reactor fluid and liquid height
  - Non -standard 4 blade pitched(45 °) blade
  - D = 38 mm
  - C = 10 mm
  - Distance between impellers = 29 mm
- **Baffles**
  - 3 x 24 mm x 25 mm
  - Pre -positioned between impellers

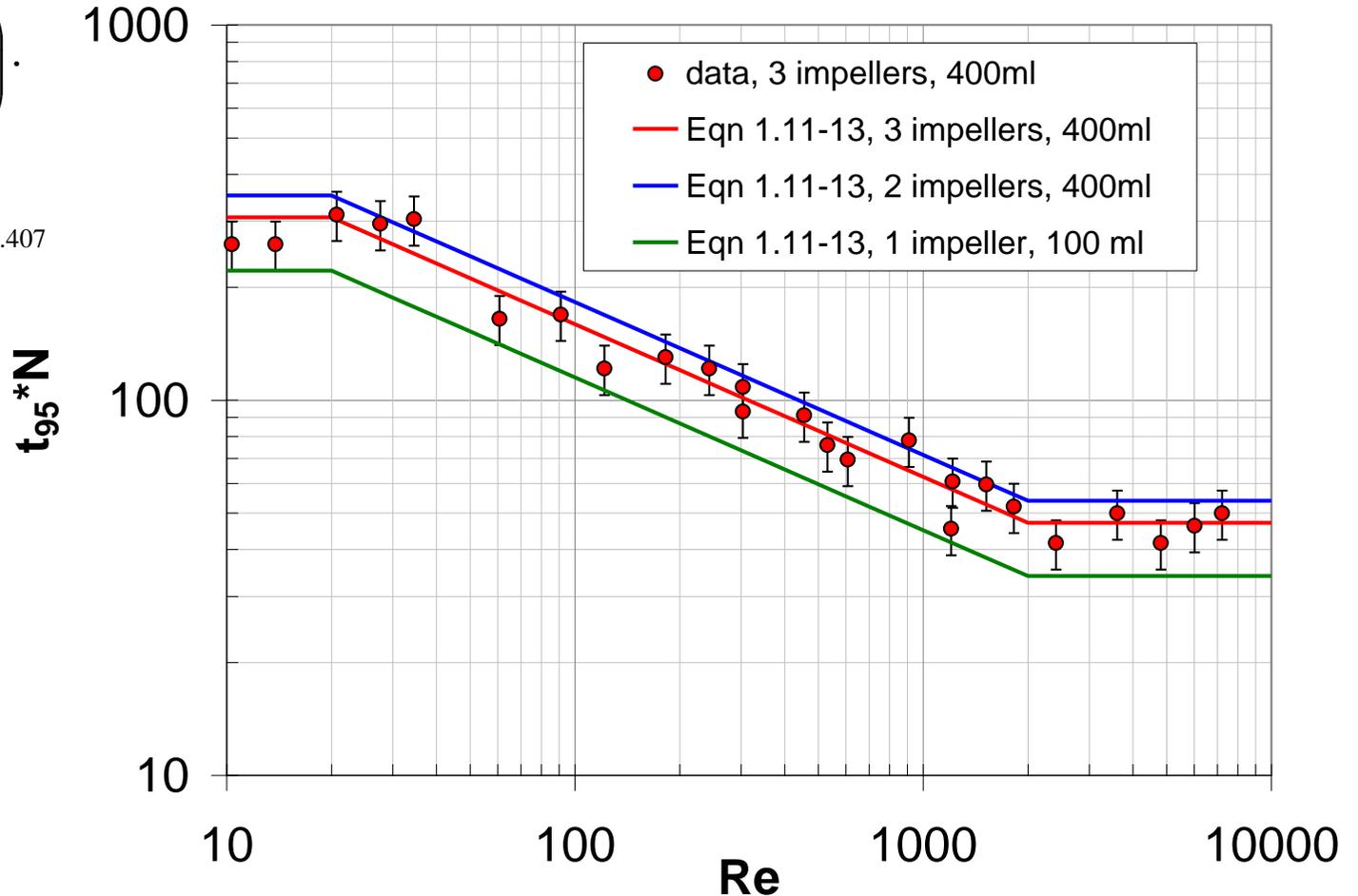
# Power curves for the AP01-0.5 were developed for 1, 2 and 3 impellers



# Macro-mixing correlations based on *Thermal Uniformity* for the AP01-0.5

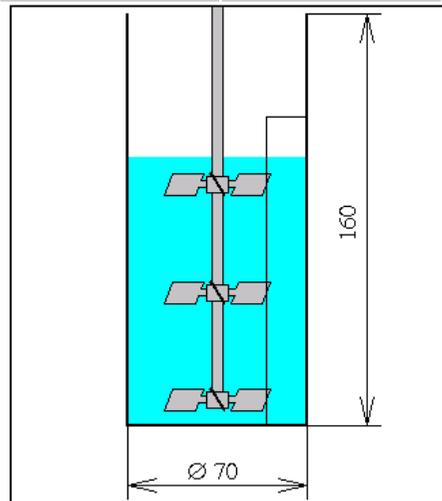
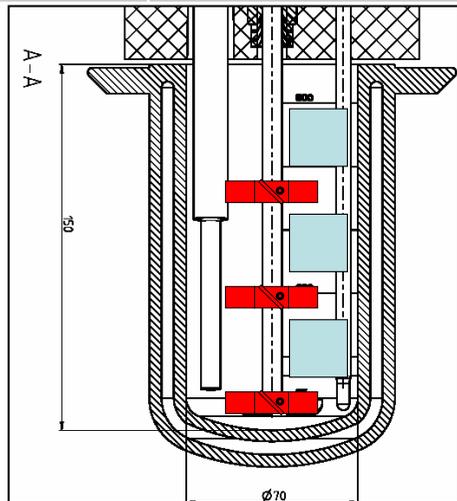
$$t_{95}(s)(\pm 15\%) = 47.1 \cdot \left( \frac{1}{N(\text{rps})} \right)$$

$$\left( \frac{3}{n} \right)^{\frac{1}{3}} \cdot \left( \frac{V(\text{ml})}{400(\text{ml})} \right)^{\frac{1}{2}} \cdot \left( \frac{2000}{\text{Re}} \right)^{0.407}$$



## Comparison of Power Numbers and “macro-mixing”

Number of impellers			1	1	2	2	3	3
	Agitation	rpm	900	300	300	120	300	100
	Re		26.0	8.7	8.7	3.5	8.7	2.9
VisiMix	$1/T_{\text{circulation}}$	1/s	0.400	0.087	0.139	0.055	0.166	0.054
Experimental	$k_{\text{mix thermal}}$	1/s	0.113	0.035	0.044	0.017	0.050	0.017
VisiMix	$k_{\text{mix composition}}$	1/s	0.0132	0.0015	0.0074	0.0026	0.0061	0.0020
VisiMix	Power number		4.5	8.7	16.3	40.2	24.3	71.0
Experimental	Power number		4.9	12.7	16.5	39.9	21.9	62.8



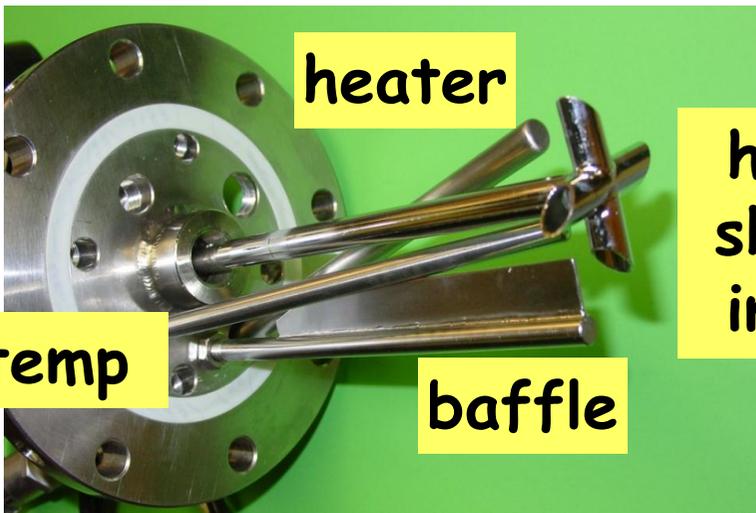
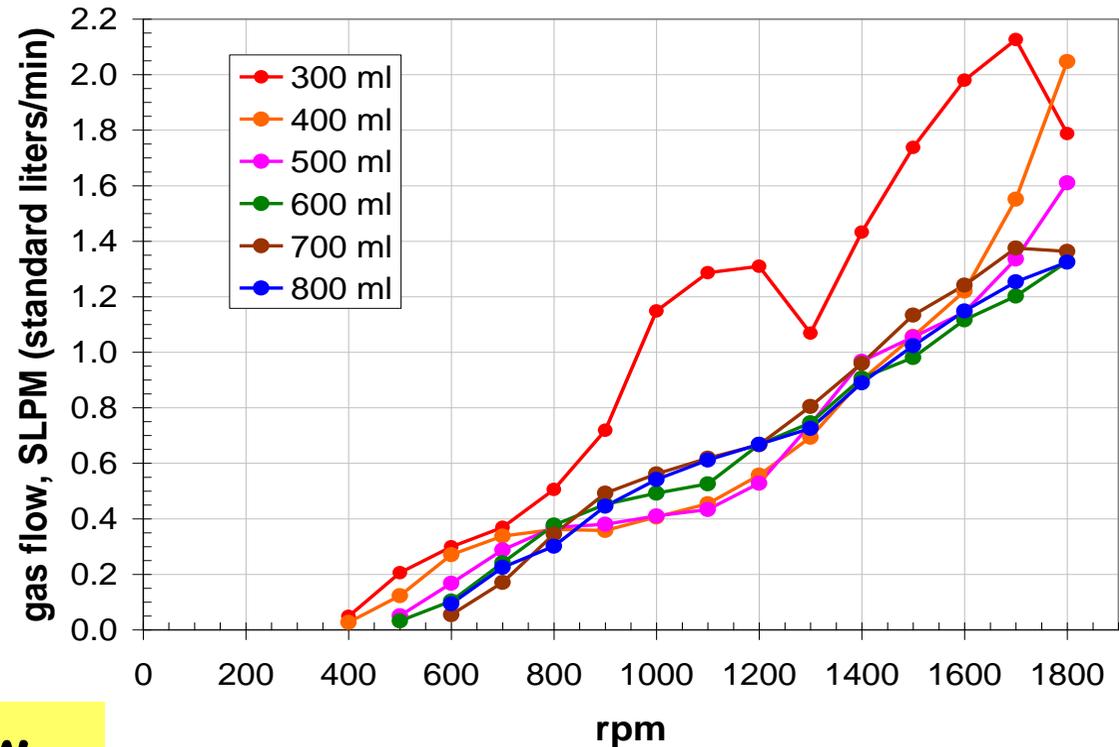
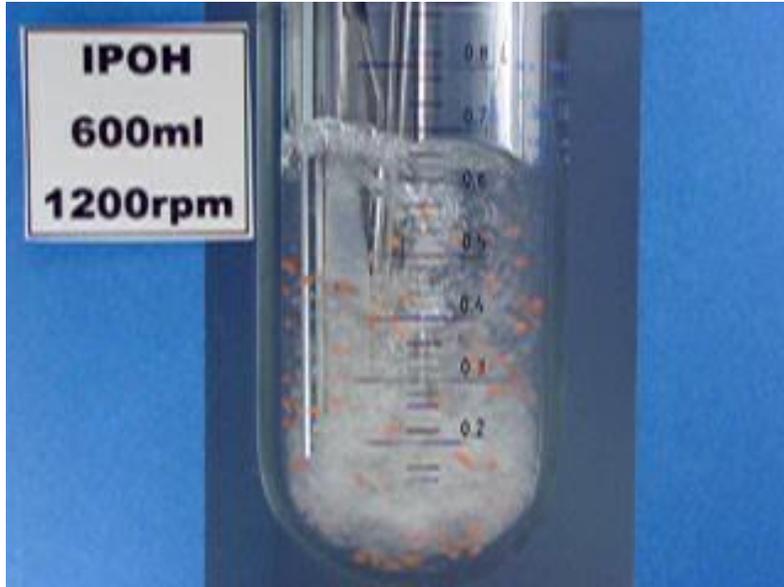
**Note that thermal uniformity in liquids is achieved much more efficiently than composition uniformity and explains in part the difference between VisiMix and the thermal tracer method.**

**$Pr \ll Sc$**

# Gas-liquid mass transfer

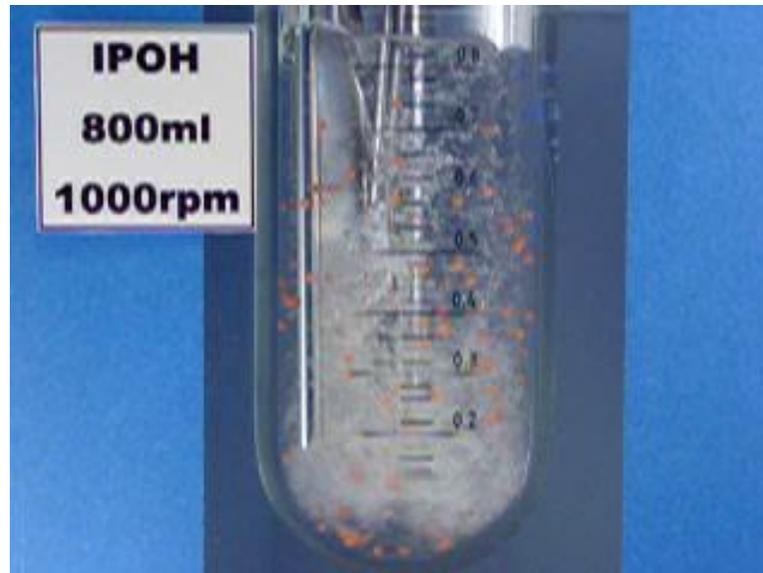
Agitation drives gas-liquid mass transfer.

RC1 study in the MP-10 reactor using the standard gassing impeller



hollow shaft & impeller

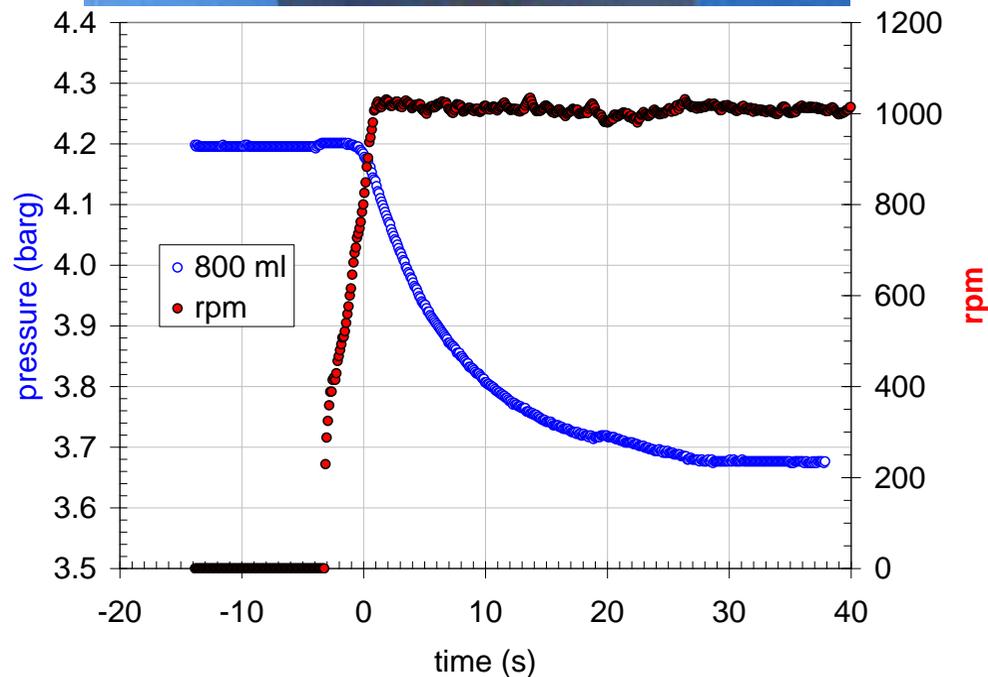
As the rpm's and impeller tip speed increase, the local pressure decreases and the gassing impeller draws gas and pumps liquid.



Rxn rate =  $k_L a$  (s<sup>-1</sup>) {  $[H_2]_{sat.} - [H_2]_{bulk}$  }

“rate constant”  
increases w/ agitation  
intensity & H<sub>2</sub> flow

“driving force”  
increases w/  
pressure



# Calculations of gas solubility and mass transfer from batch absorption

$$C_{H_2,sat} = (P_M - P_F) \cdot \frac{V_G}{V_L} \cdot \frac{1}{RT}$$

$$\ln \left\{ \frac{P - P_F}{P_M - P_F} \right\} = -k_L a \cdot \left\{ \frac{P_M - P_E}{P_F - P_E} \right\} \cdot t$$

$k_L a$  = Mass transfer coefficient

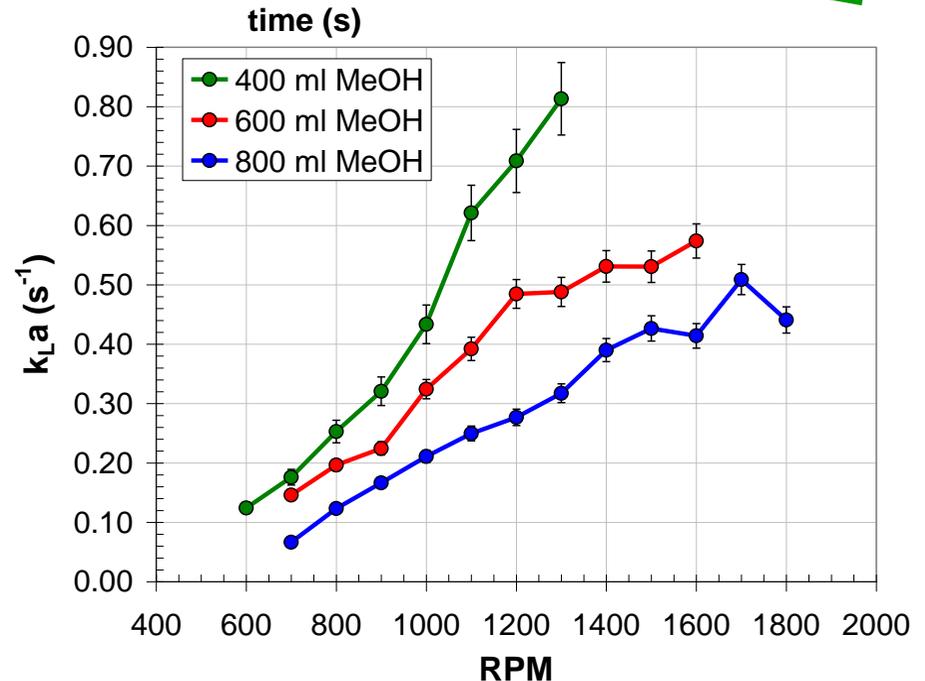
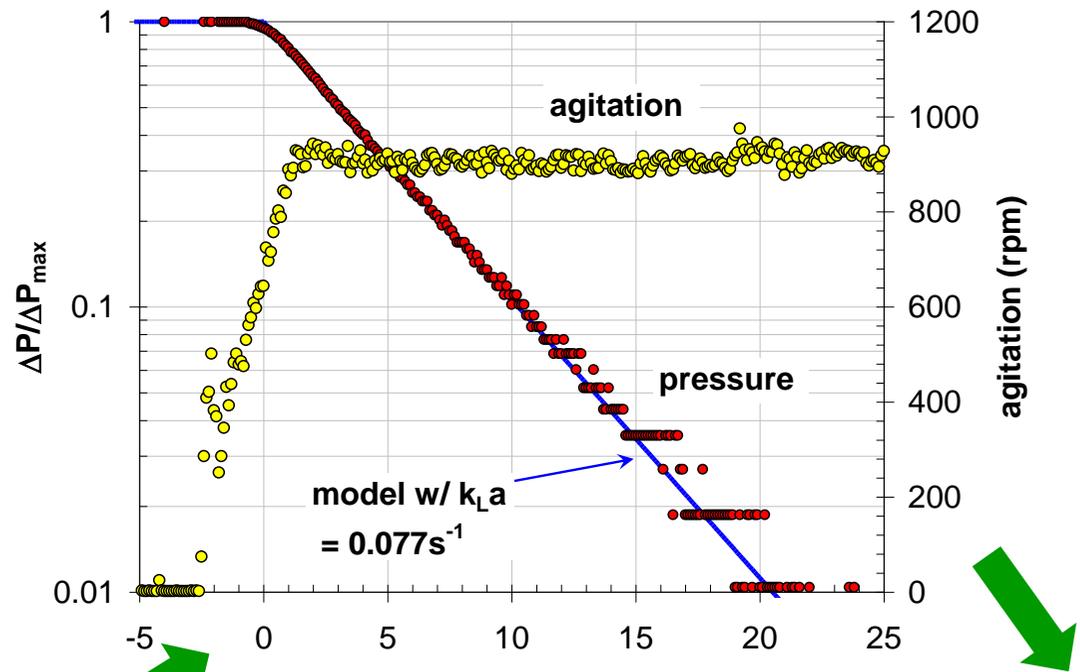
$P$  = Pressure as a function of time;

$P_M$  = Maximum pressure from initial gas charge

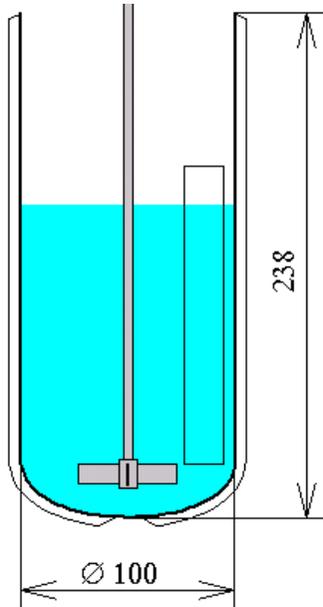
$P_E$  = Initial equilibrium pressure with no gas added;

$P_F$  = Final pressure

$V_G$  = Head space gas volume;  $V_L$  = Liquid volume



# Characterization of lab impellers key to use of models: VisiMix flat turbine impeller width adjusted to match Power number of the lab agitator

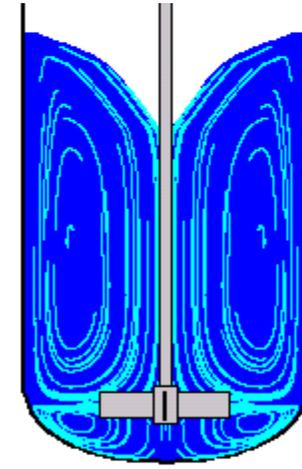
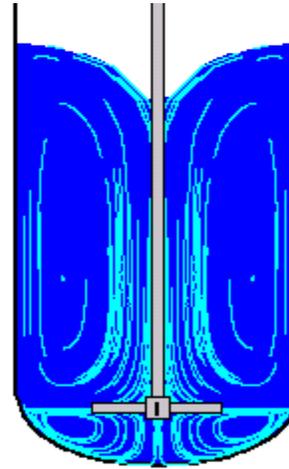


VisiMix model of HP60 w/ single baffle; gassing impeller is modeled as a flat blade turbine With  $N_p=1.7$ .

Experimental values for HP60, T=10 cm; standard gassing impeller D=4.7mm, W=10mm; fluid at 140°C, $V_L=1.089$ liters, surface tension=26 dyne/cm, viscosity=0.25 cP, density=930 gm/liter.				VisiMix estimate w/ impeller power matched to experiment (blade width = 4.4 mm power number $N_p=1.7$ )		VisiMix estimate w/ true impeller blade width =10 mm power number $N_p=3.5$	
	super- ficial velocity	agitator power	H <sub>2</sub> mass transfer coeff., $k_L a$	agitator power	H <sub>2</sub> mass transfer coeff., $k_L a$	agitator power	H <sub>2</sub> mass transfer coeff., $k_L a$
rpm	cm/s	W/kg	1/s	W/kg	1/s	W/kg	1/s
800	0.16	0.8	0.15	0.9	0.029	1.8	0.066
1000	0.23	1.6	0.23	1.7	0.074	3.5	0.18
1200	0.31	2.7	0.33	2.9	0.16	6.0	0.40
1400	0.49	4.6	0.42	4.6	0.33	9.6	0.87

# Summary of differences in model approximations for VisiMix at 1400 rpm

measured  $k_L a = 0.42 \text{ s}^{-1}$   
@4.6 W/kg



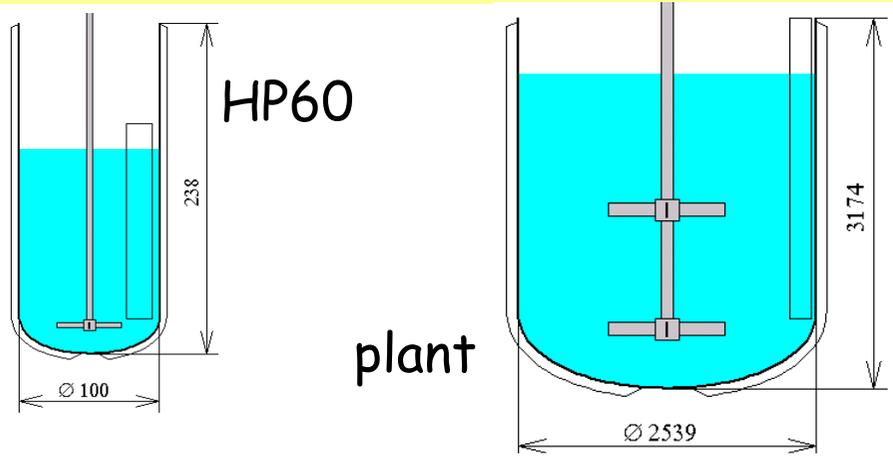
Blade width, mm	4.4 (flat blade turbine adjusted to match measured power)	10 (actual width of impeller)
$k_L a, \text{ s}^{-1}$	0.33 (exp. = 0.42)	0.87
vortex depth, cm	2.1 (exp. vortex ~2.5 cm)	3.6
average energy dissipation, W/kg	4.6	9.5
gas hold-up	3.5%	5.5%
Sauter mean bubble, mm	1.8	1.4
circulation rate, liters/s	1.7	2.4
micro-mixing time, s	0.14	0.088
circulation time, s	0.64	0.46

## What about the scale-up of mass transfer?

- plant & lab raw materials & catalyst are from same lots numbers
- mixing in lab reactor measured and  $k_L a > 0.4 \text{ s}^{-1}$ ; mass transfer is very fast compared to reaction so that  $C_{\text{H}_2, \text{bulk lab}} = C_{\text{H}_2, \text{sat lab}}$
- lab reactor pressure was adjusted to match plant rate profile
  - RC1 programmed to match exact temperature profiles of plant
  - when plant = 800 psig and lab = 700 psig the rates are equivalent

$$C_{\text{H}_2 \text{ bulk lab}} = C_{\text{H}_2 \text{ saturated lab}} = C_{\text{H}_2 \text{ bulk plant}}$$

$$k_L a_{\text{plant}} = \frac{\text{rate}}{(C_{\text{H}_2 \text{ saturated plant}} - C_{\text{H}_2 \text{ saturated lab}})}$$

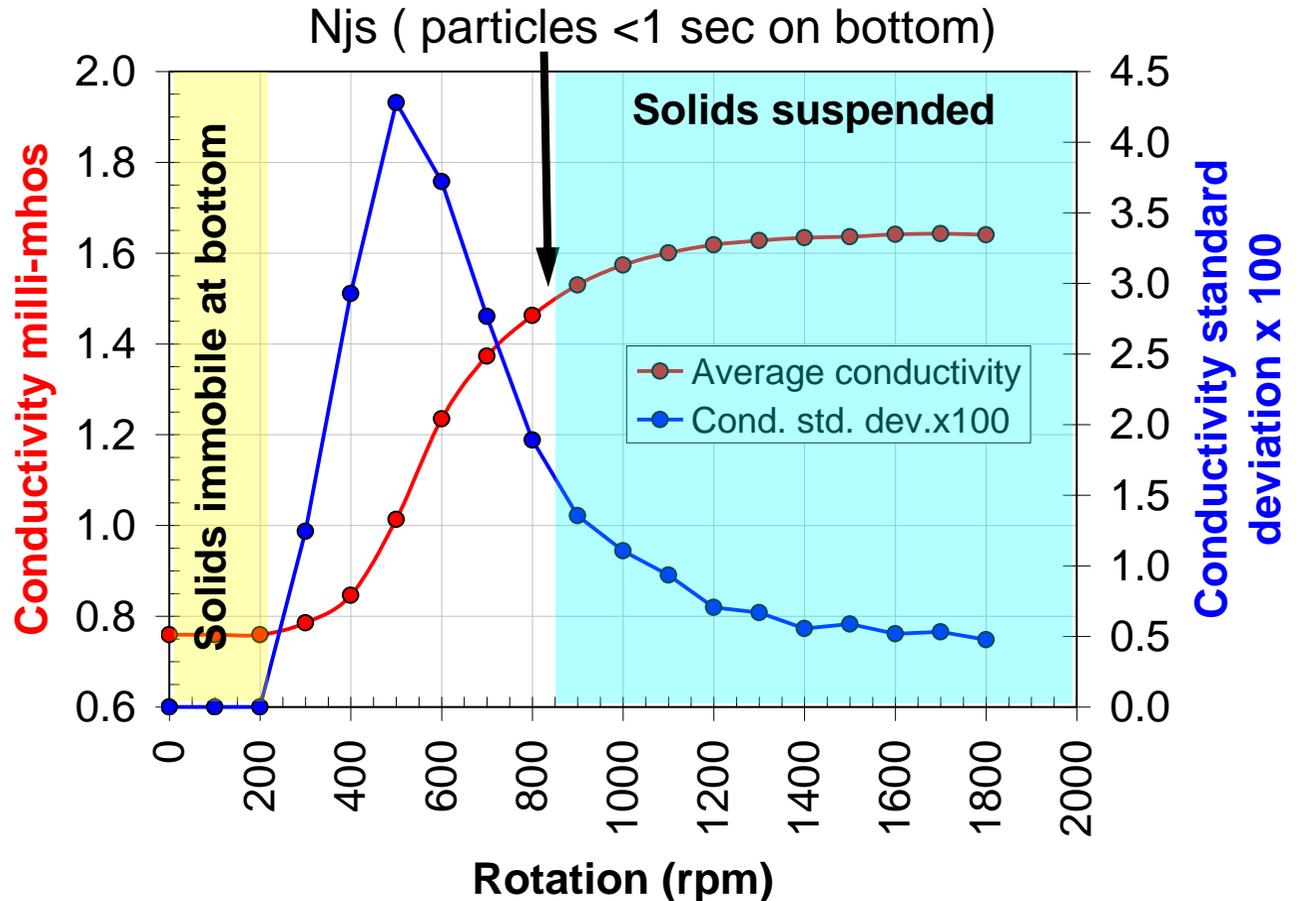
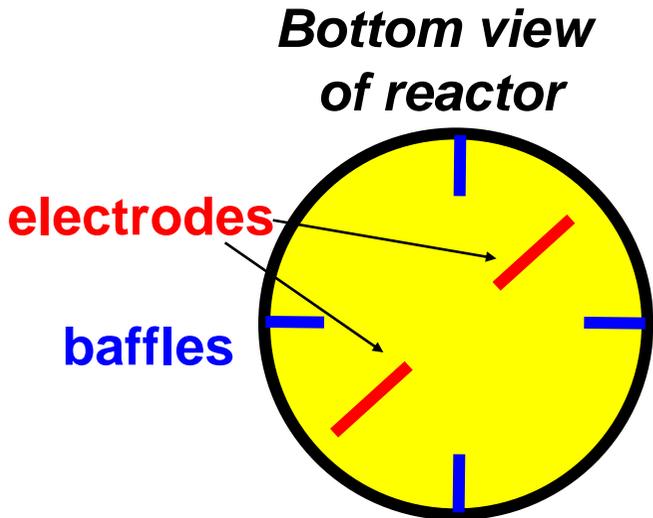


	plant	RC1 HP60
volume	3300 gal	1.0 liter
agitation	84 rpm	1400 rpm
Impeller diameter type	40 inch 2x flat turbines	1.8 inch gassing
pressure	800 psig	700 psig
$k_L a$ from scale-down	0.052 s <sup>-1</sup>	
$k_L a$ from VisiMix	0.062 s <sup>-1</sup>	

## Agitation intensity impacts solid suspension and solid-liquid mass transfer

- Suspension occurs when particle kinetic energy is greater than its gravitational energy.
- Mass transfer is optimal when solids are suspended off the bottom of the vessel.

The  $N_{js}$  (*rotation rate of just suspension*) is determined by observing solids on the bottom of a vessel and is the agitation rate when any solid particle spends less than 1 second on the bottom of the reactor.



# Classic Zweitering correlation characterizes solids suspension

- Zweitering, T.N. *Chem. Eng. Sci.* (8) 1958, 244

$$N_{js} = \frac{S \cdot \nu^{0.1} \cdot d_p^{0.2} \cdot \left( g \cdot \frac{\Delta\rho}{\rho} \right)^{0.45} \cdot X^{0.13}}{D^{0.85}}$$

$N_{js}$  = Critical rotation rate, rps, to achieve suspension

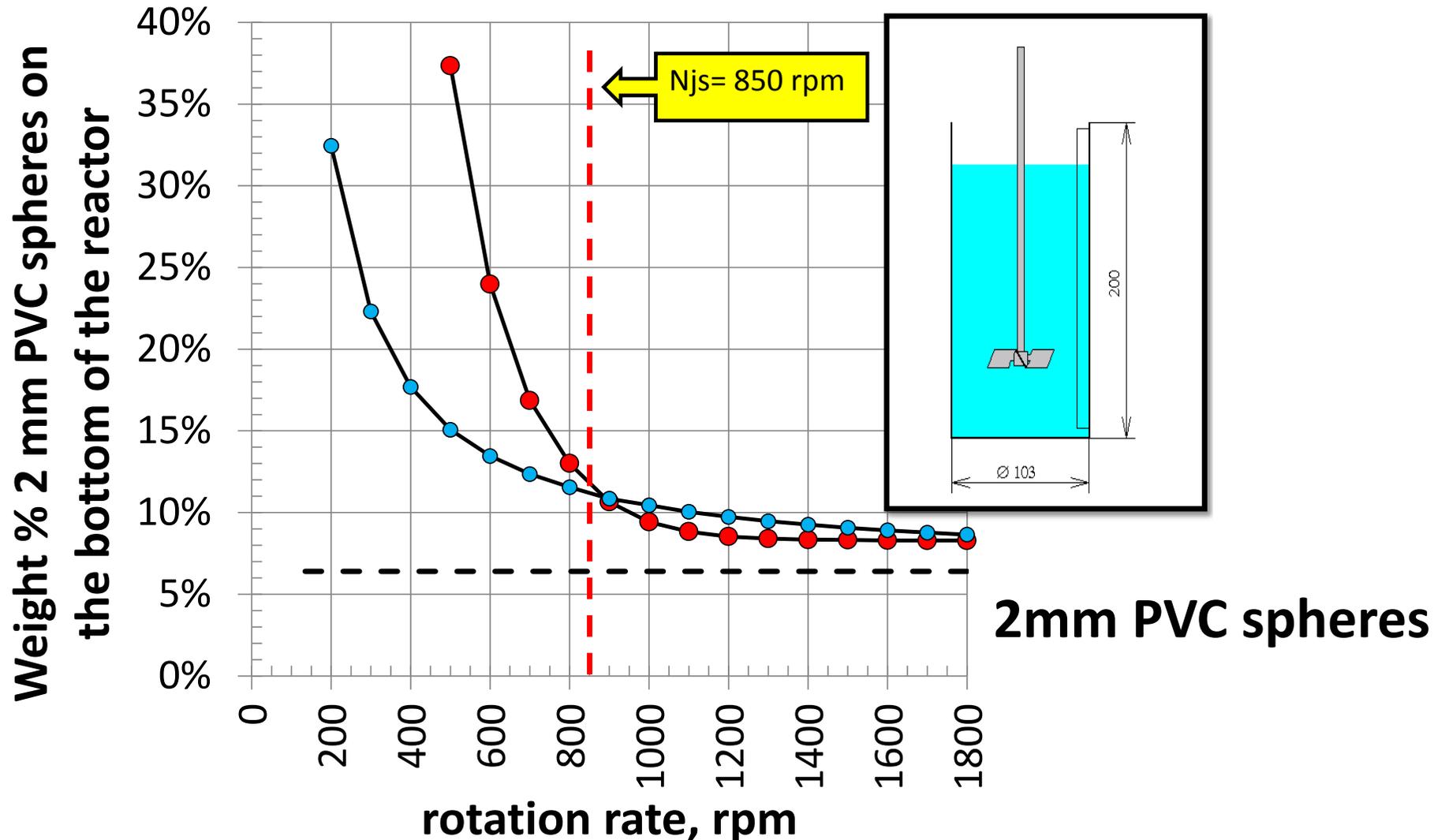
$S$  = Impeller Constant, Ranging from 2 to 20       $\nu$  = Kinematic viscosity, m<sup>2</sup>/s

$d_p$  = Particle size, m       $g$  = gravitational constant, 9.8 m/s<sup>2</sup>

$\Delta\rho$  = Particle density - Liquid density, kg/m<sup>3</sup>       $\rho$  = Liquid density, kg/m<sup>3</sup>

$X$  = Weight solid/weight liquid \* 100       $D$  = Impeller diameter, m

# Comparison between predicted and estimated (by conductivity) solids concentration at the bottom of a lab reactor



## Scale-up of the solid liquid mixing based on the Zweitering correlation

If we assume that the composition and physical properties are the same in the lab and the plant.

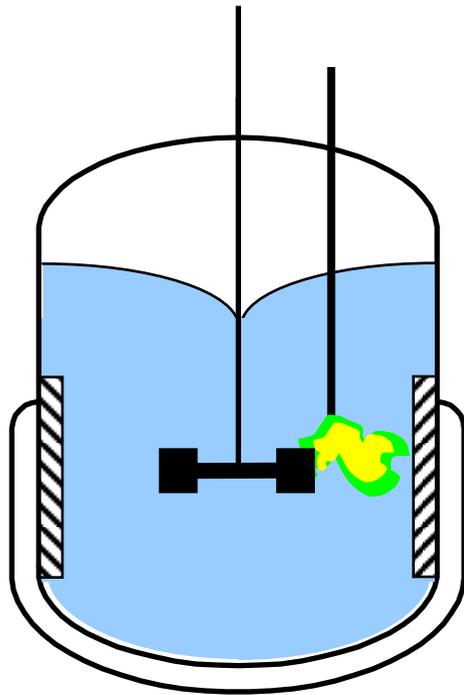
$$\frac{N_{js,Plant}}{N_{js,Lab}} = \frac{\left\{ \frac{S_{Plant}}{D_{Plant}^{0.85}} \right\}}{\left\{ \frac{S_{Lab}}{D_{Lab}^{0.85}} \right\}} = \left( \frac{S_{Plant}}{S_{Lab}} \right) \cdot \left( \frac{D_{Lab}}{D_{Plant}} \right)^{0.85}$$

## Scale-up of the solid liquid mixing using VisiMix

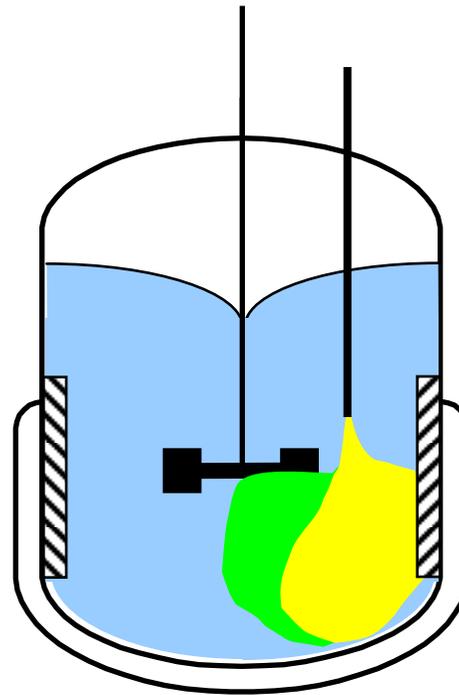
- Match solids concentration profiles between lab and plant
- Insure “particle-particle” energy in plant reactor  $\leq$  lab reactor (lower solids may be necessary)

**Note that I was not successful in matching solid hold-up between VisiMix and lab reactors unless the exact geometry of the lab reactor was matched with the VisiMix reactor geometry selections.**

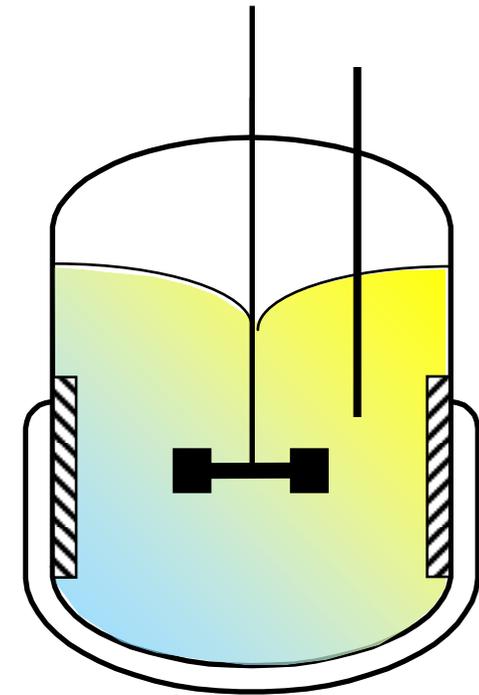
## Visualizing reaction regimes in a turbulent low viscosity system for a fast reaction



micro-mixing



meso-mixing



macro-mixing

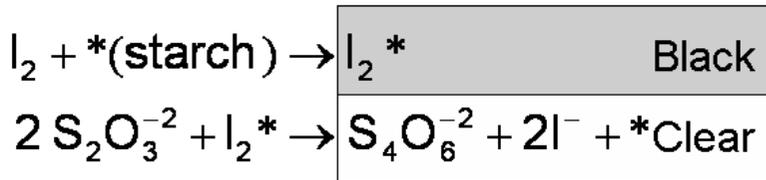
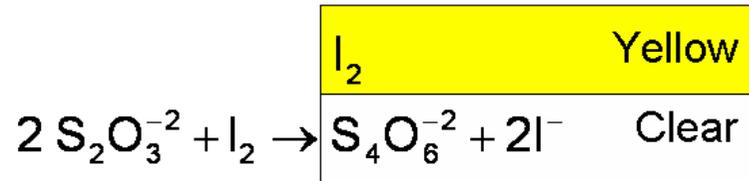
## Different mixing mechanisms are impacted differently by feed rate, feed location, viscosity and the number of feed points

mechanism	Dependency of mechanism				Rule-of-Thumb Scale-up variable to keep constant
	Feed tube location	Feed rate	Viscosity	Number of feed points	
Micro	Yes	No	Yes	No	$P/V$ or $N^3D^5/V$
Meso	Yes	Yes	No	Yes	$nN^4D^3/Q_{\text{feed}}$
Macro	No	Yes	No	No	$ND^3/Q_{\text{feed}}$

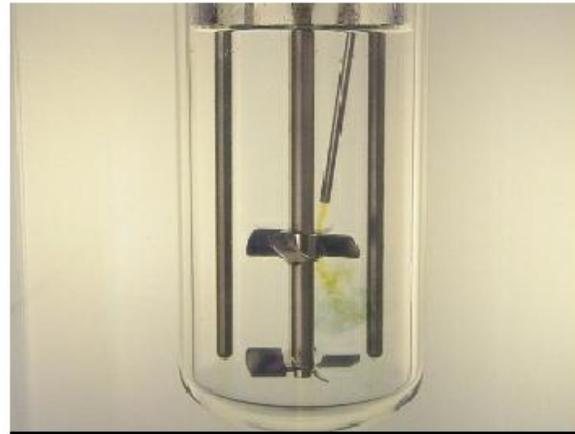
Semi-batch addition of I<sub>2</sub> solution added to S<sub>4</sub>O<sub>6</sub><sup>2-</sup> and starch.

AP01-0.5 reactor, 400 cc

Process chemistry Final color



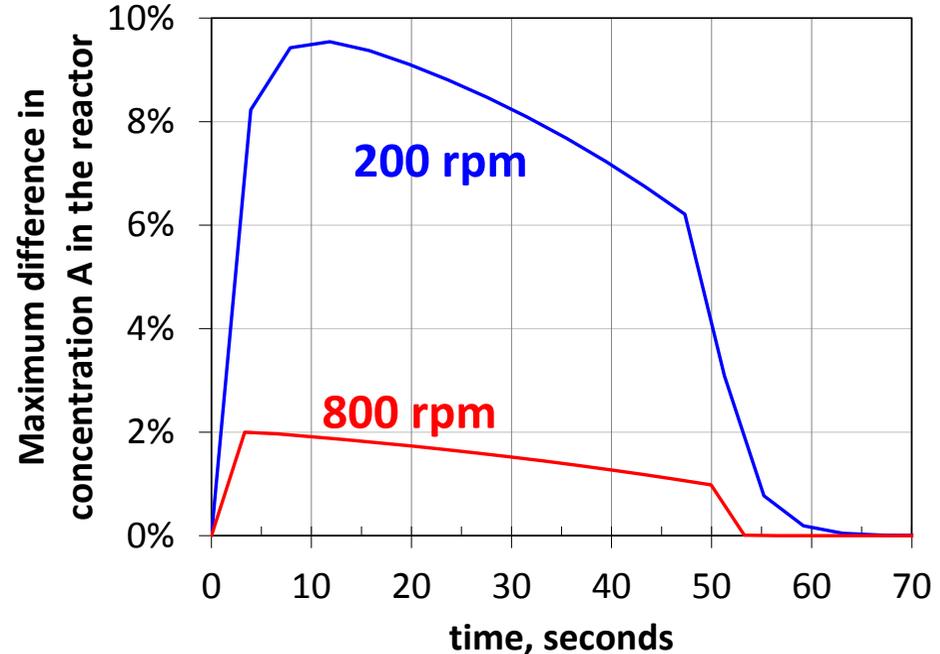
VisiMix prediction of maximum difference in thiosulfate solution during addition of I<sub>2</sub> solution. (rate constant estimated)



200 rpm

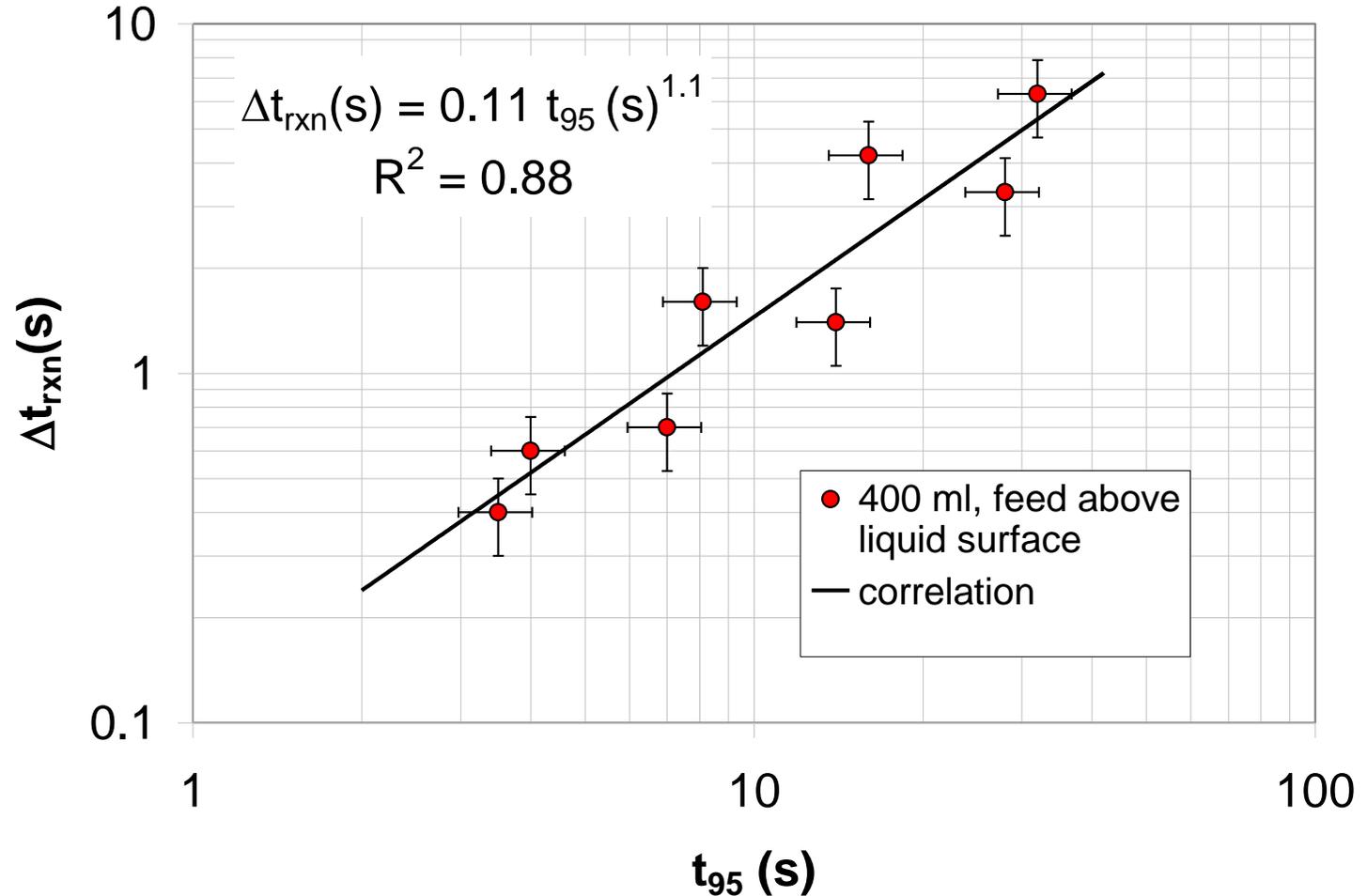


800 rpm



## Correlation of macro-mixing time with the reaction time for the iodine-thiosulfate reaction system.

The reaction time for the iodine/starch black color to disappear,  $\Delta t_{\text{rxn}}(s)$ , is measured from the time the feed is stopped to the time the solution is visually 100% clear.



## So what have I/we learned?

- **VisiMix predictions and lab reactors mixing measurements agree well when reactor geometries match and physical properties are known.**
- **If lab reactor geometries do not match the catalogue of available geometries in VisiMix, geometric approximations must be made.**
  - **For power and heat transfer, errors appear to be insignificant.**
  - **For solid suspension, phase dispersion and circulation**
    - **more detailed mixing data for the lab reactor is necessary to adjust the approximations.**
    - **validations may be necessary.**
- **The limitations of lab mixing characterization methods such as thermal tracing must be considered when using them for scale-up or matching more detailed VisiMix models.**

And now for some practical advice...

If a semi-batch process gives you poor results on scale-up.....

**SLOW DOWN THE FEED RATE**



**Dilute the Feed Concentration**

**Increase mixing intensity**



**Lower the temperature**

## **Application of Visimix to the Characterization of Lab Reactors**

**A critical step in the effective scale-up of a laboratory stirred tank process is understanding the mixing parameters at the lab scale and then the anticipating the role of mixing at different scales. This fundamental understanding requires detailed mixing characterization of the laboratory reactor. Independent measurements of the power and macro-mixing times were made in various lab reactors and compared to estimates made by the VisiMix mixing tool. Heat and mass transfer coefficients where measured and compared to predictions made using VisiMix. The measurements and calculations compare very well, validating the VisiMix methodology.**

## Contact information

Reinaldo “Ray” Machado

phone: (484) 553-3612

E-mail: [info@rm2tech.com](mailto:info@rm2tech.com)

Website: [www.rm2tech.com](http://www.rm2tech.com)

**Ray is the instructor of short course  
“Fundamentals of Scale-up”, which may be offered at your site.**



Reinaldo (Ray) Machado is the developer and instructor of a popular industrial short course, “Fundamentals of Scale-up” which he teaches part time. He is currently employed by Air Products and Chemicals, Inc. in Allentown, Pa. in 1986 and serves as a senior consultant specializing in reaction engineering. Previously, he managed hydrogenation and oxidation applications development for the Catalytic Reaction Solutions Group and served as group leader for the Reaction Engineering Technology area. Ray has broad technical experience in applied reactor engineering, scale-up of chemical reaction processes, mass transfer, heat transfer, applied reaction calorimetry, hydrogenation, electrochemical engineering, sulfonation, amination, propoxylation, polymerization, and plastics recycling.

Ray received a Ph.D. in chemical engineering with a concentration in chemistry from the University of Wisconsin, Madison, and a B.A. in chemistry and mathematics from Frostburg State College. He has served as a part-time instructor of a short course, “Scale-Up Considerations in Chemical Processes,” at Lehigh University and currently teaches industrial courses on the fundamentals of scale-up. He holds 16 patents, has collaborated on 15 publications, and is a member of the American Institute of Chemical Engineers and the American Chemical Society.



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