Methodological Aspects of On-Line Probes in Stirred Tanks

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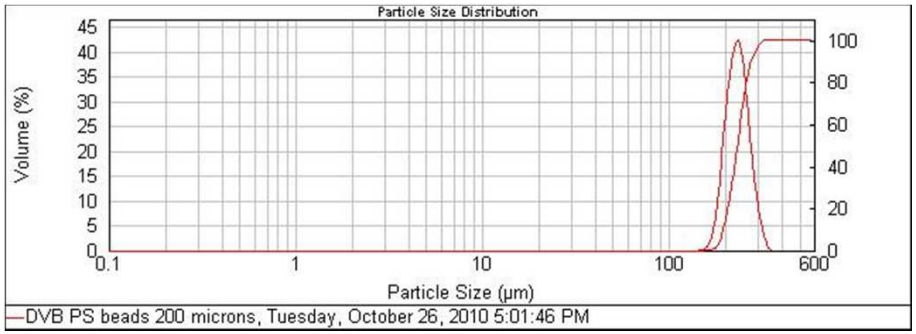
Objectives and Outline

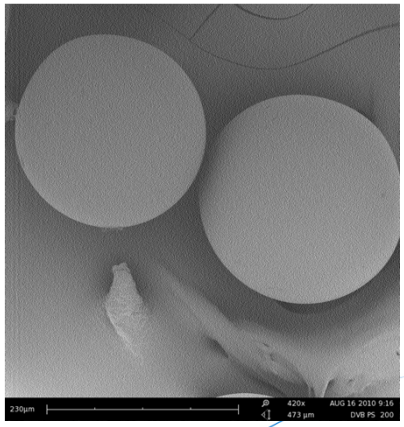
- *Overall objective of the project: develop understanding of contribution of reactors hydrodynamics to data* from on-line probes (FBRM, Raman, IR…)
  - Study of impact of hydrodynamics on response of FBRM measurements in stirred tank reactors
- **Outline**
  - Experimental:
    - Materials, Instruments, Techniques
      - Model solid particles
    - Impact of stirring speed and probe location on size distribution
      - Total number of counts
      - False bimodality
  - Approach to data analysis
    - Hydrodynamic simulation of reactor: distribution of solid particles
  - Future plans

Materials, Instruments, Techniques

- **Instruments & Techniques**
  - **Glass reactor**
    - 3L; Ø150 mm
    - 3-blade pitched stirrer (50-600 rpm)
- **Particles Characterization**
  - **SEM FEI Phenom**
  - **Mastersizer 2000 (Malvern)**
    - Dry module, 3 Bars
  - **FBRM D600 (Mettler Toledo)**
    - Laser beam rotation at 2m/s
    - 1-1000 microns
- **Reactor simulation software**
  - VisiMix®
    - Turbulent 2K package

- **Materials**
  - **Polymer beads (DVB/PS; PVC)**
  - **De-mineralized water**





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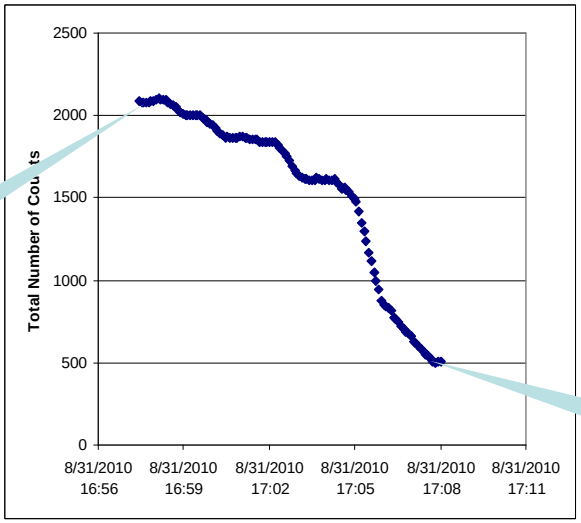
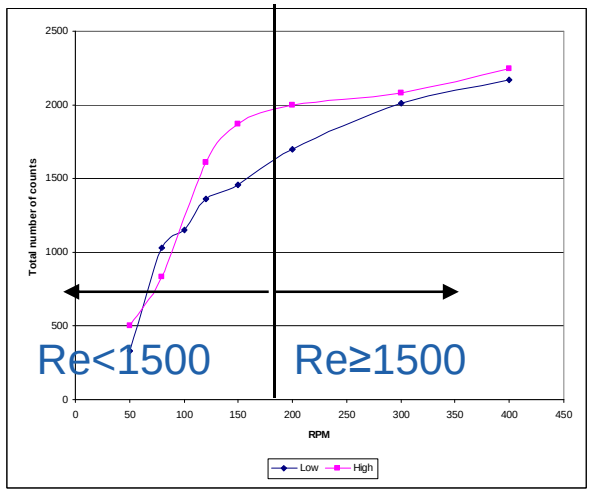
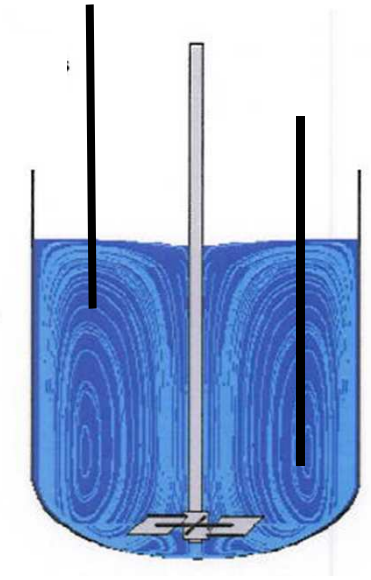
Experimental: Total number of Counts and Stirring Speed





High position (100 mm from the bottom)



Low position (20 mm from the bottom)

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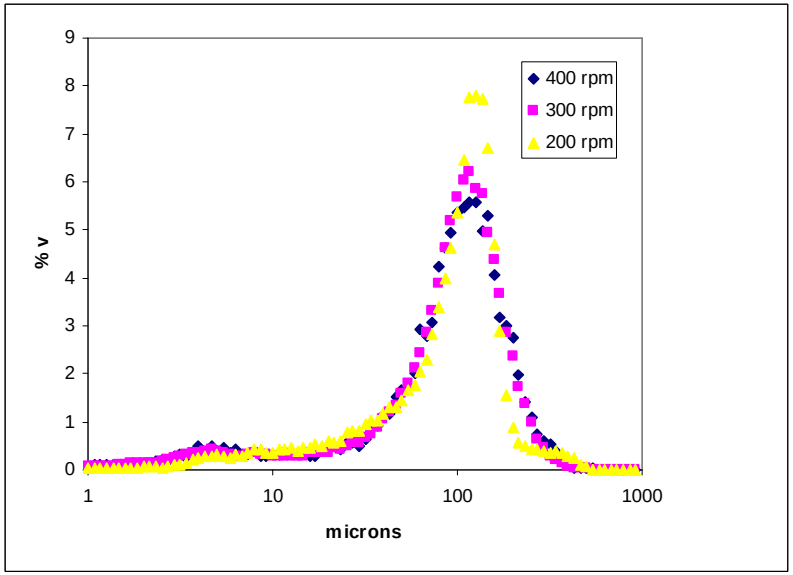
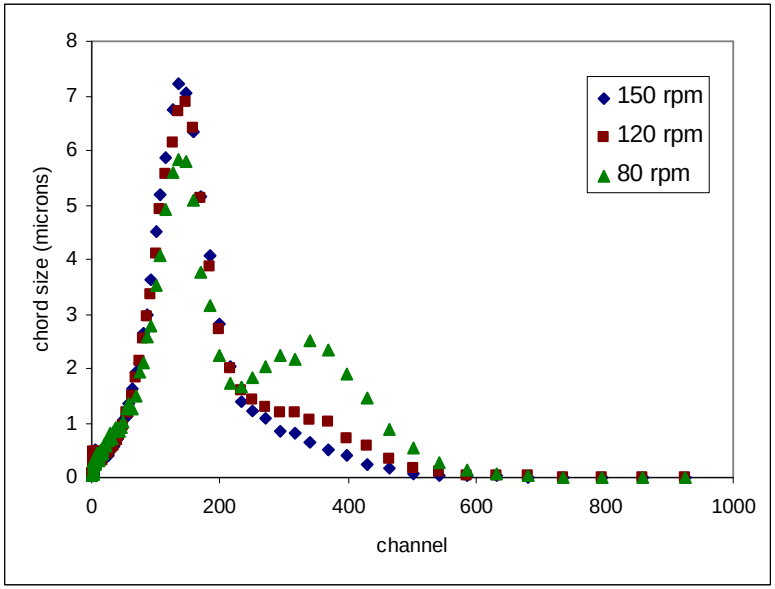
Experimental: Stirring and Chords Size Distribution



PVC 100 microns

Re>1500

Low probe position



DVB/PS 200 microns

Re <1500

Low probe position

Summary of Experimental Observations

- **FBRM readings are affected by**
  - **Location of the probe**
  - **Stirring speed**
- **Impact on ~~particle~~ (chords) size distribution**
  - **Total number of counts depends on position of the probe and stirring speed**
  - **Low stirring speed can lead to identification of false bi-modality**

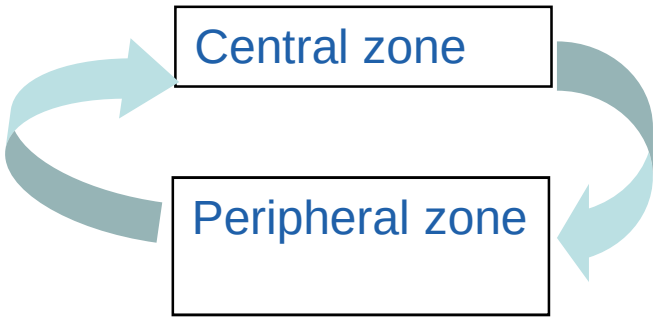
FBRM data reflect variation of the concentration of particles in the reactor.

What factors effect variation of size distribution over the reactor volume?

Hydrodynamic Simulation (VisiMix® Approach)

- **Background**
  - Two-zone model of reactor
  - Governing equation: moments balance (impeller/liquid/reactor walls)
  - Approximation of flow rate by power series

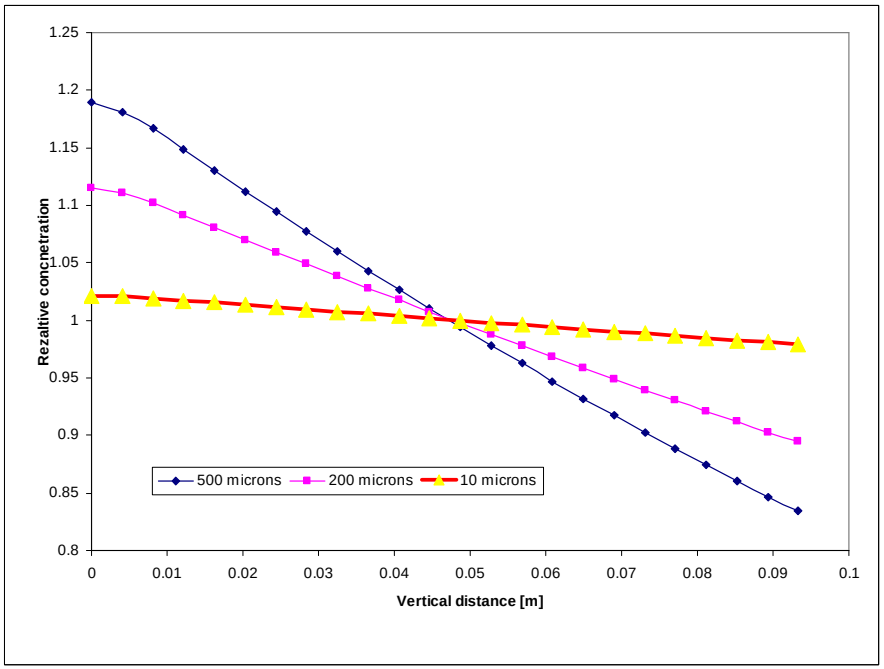
- **Assumptions & Limitations**
  - 1500 < Re < 2,000,000 Turbulent flow
  - Radial flow profile does not depend on vertical coordinate
  - Cylindrical geometry of peripheral and central zones
  - Solid particles flow with the same rate as liquid
  - The model includes some empirical coefficients- validity of VisiMix is limited by validity of empirical interrelations



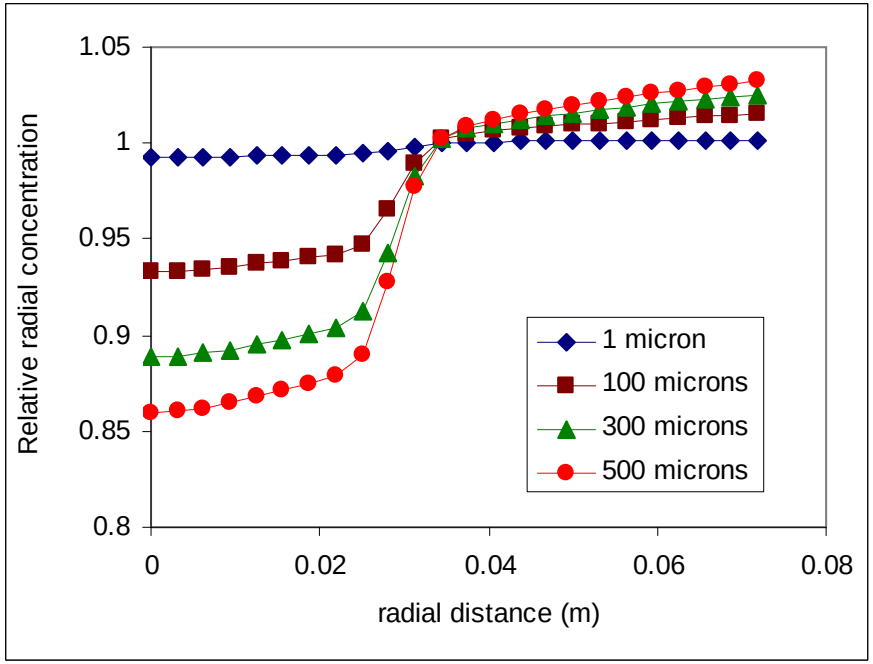
Structure of two zone model of stirred tank

VisiMix Simulation: Stirring Speed & Particles Distribution in 3 L reactor (DVB PS particles)

Axial Distribution (500 rpm)



Radial Distribution (500 rpm)



Axial and radial distributions vary with particles size: impact on PSD is expected

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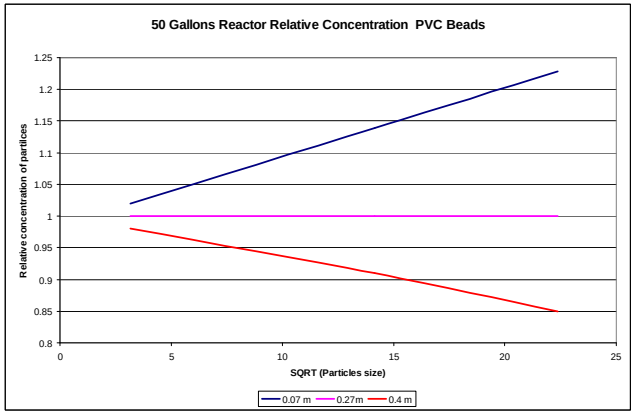
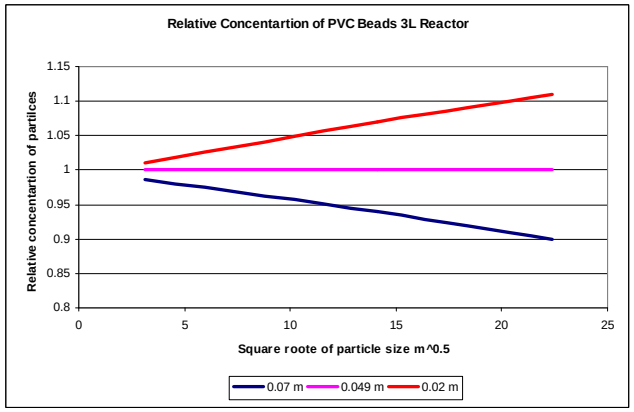
Summary of Hydrodynamic Simulation

- **Probe location**
  - ~90% of particles are located in peripheral zones of stirred tank *(for heavy particles)*
  - Within the peripheral zone slight effect of radial position of the probe
  - Impact of axial probe position increases with particles size and weight
- **Assessment of hydrodynamics impact on size distributions by FBRM**
  - For each size class of particles calculate local hydrodynamic factor i.e. ratio of local particles concentration to average
    - Density of phases, design of reactor, viscosity should be known
    - $N^{J}_{FBRM} = N^{J}_{true}\, n^{J}(r, h)$





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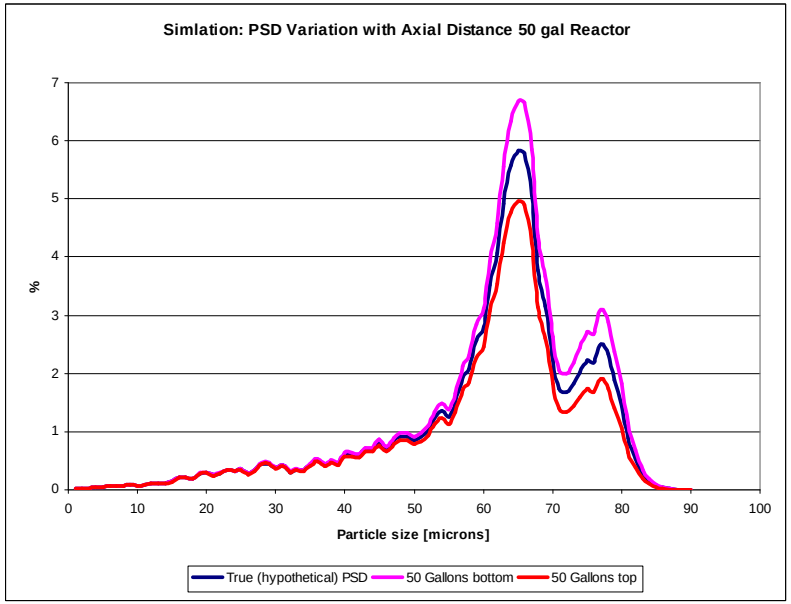
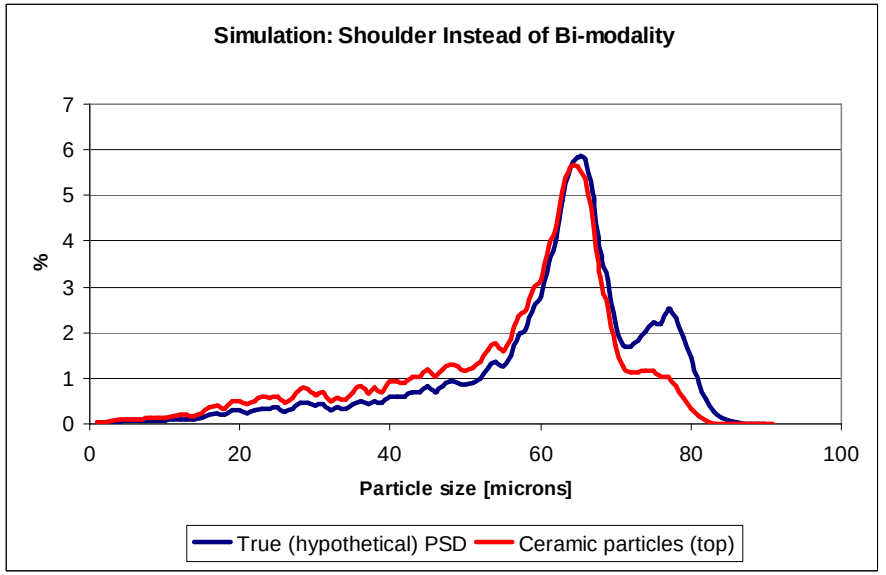
VisiMix Simulation: PSD Variation





PSD Variation: DVB/PS particles

PSD Variation: Ceramic particles

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Conclusions and Further Plans

- **Impact of hydrodynamics of stirred tanks on FBRM was shown experimentally**
  - **Stirring speed**
  - **Probe location**
- **Results of simulation show potential impact on sizing and size distribution**
- **Correct interpretation of on-line probes data requires**
  - **Measurements at rpm providing uniform distribution of particles-*if possible***
  - **Optimal probe placing - *if* "no-effect-on- PSD point" *exists***
  - **Understanding of hydrodynamics**
    - Application of VisiMix or other hydrodynamic simulation tools is useful
- **Further work**
  - **Experimental verification for wide and bimodal PSD; concentration effects**
  - **Application to real products**
  - **Do we really have preferable (no-effect-on- PSD-point) probe location?**
  - **Extension of VisiMix® application to other online probes**