M7
SOLIDS LIQUID MIXING

OFF BOTTOM AND
DISTRIBUTION AND ATTRITION

SOLID-LIQUID MIXING

- **Solid suspension**
- **Solid distribution**
- **Dual impeller configurations**
- **Jet solid suspension**
- **Attrition - breakage**

PROCESS RESULTS

- Off bottom suspension
- Distribution vertically (and horizontally)

- Secondary
- Size reduction – break up of agglomerates
- Draw down of floating solids
  - Wetting
  - Low solids density
  - Low bulk density – dispersion

- NOTE: dispersion often has a double meaning
  - Dispersion over height and break up or dispersion of primary particles

****CAUTION****

- There are two kinds of slurry.
- <u>Slow Settling:</u> Pseudo-homogeneous.
  Treat as non-Newtonian.
  High concentration.
  Small particles.
- <u>Fast Settling:</u> Rate > 1 m / min.
  Two phases (solid and liquid).
  Low concentration.
  Large particles.
- Need to observe sample.

Solid liquid mixing

- Most common mixing application
- The problem with solids in processes
  - 90 % of “fluid only” plants achieve project goals
  - 60 % of solid handling plants achieve project goals
  - ~ 3 months start up time for a “fluid only”;
  - 9- 18 months for a solids handling plant

Background

- In DuPont, 80% of products are particulate solids or have solids handling steps in their process.

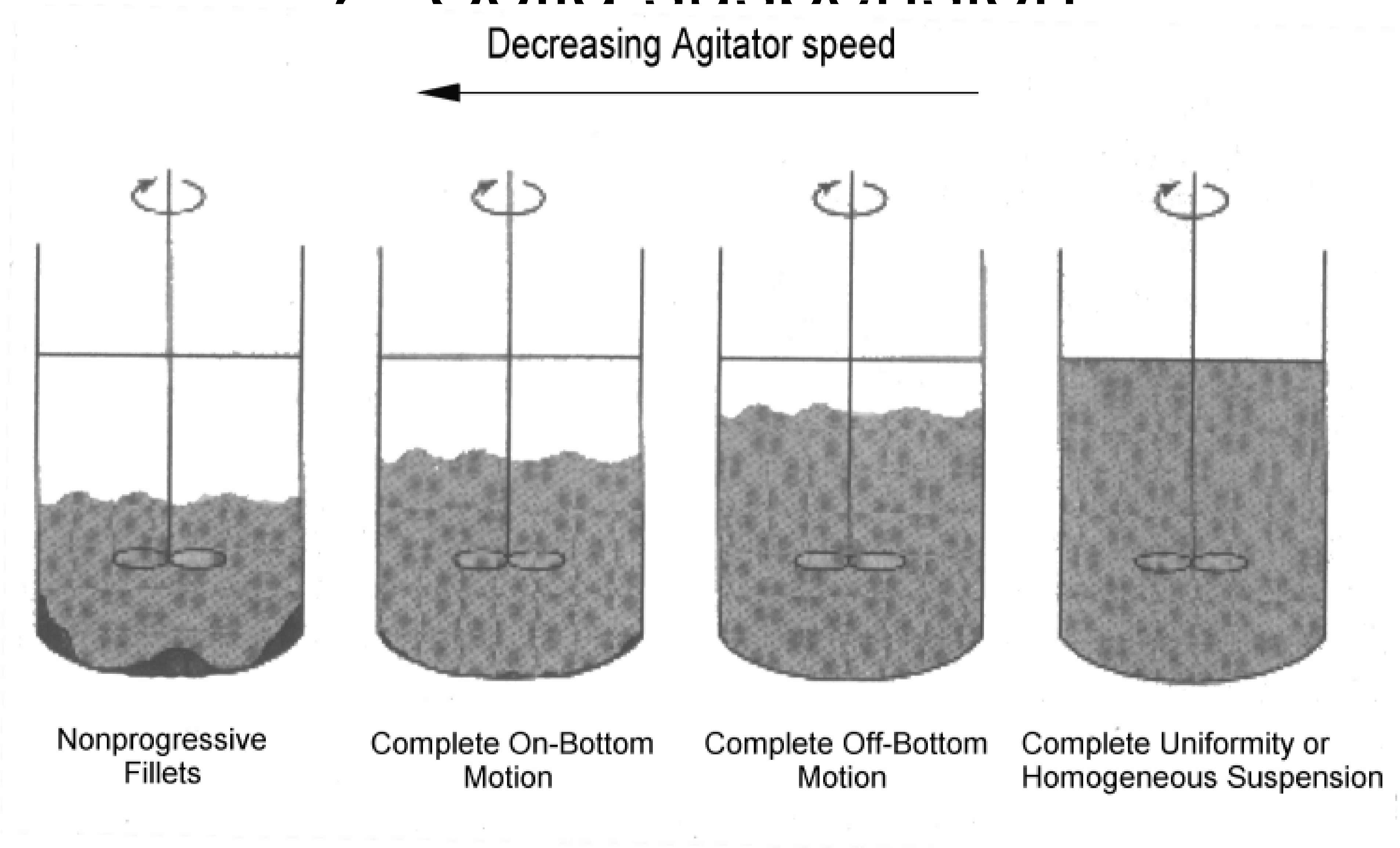
- Plants which have solids handling steps are most likely to experience problems on start-up.

- Examples of problems:
  - Slurry transport in pipelines – plugging when particles settle out:
    - Insufficient velocity.
    - Poorly designed piping - elbows, valves etc.
  - Particle attrition in equipment - separation problems.
  - Poor mass transfer rate:
    - Settled particles in reactors (e.g. catalyst).
  - Etc. etc.

SOLID LIQUID MIXING

- Lots of good data
- Hard to correlate
- Correlations not readily obvious
- Three process results
  - Off bottom
  - Mass transfer
  - Vertical distribution

2. Solid suspension



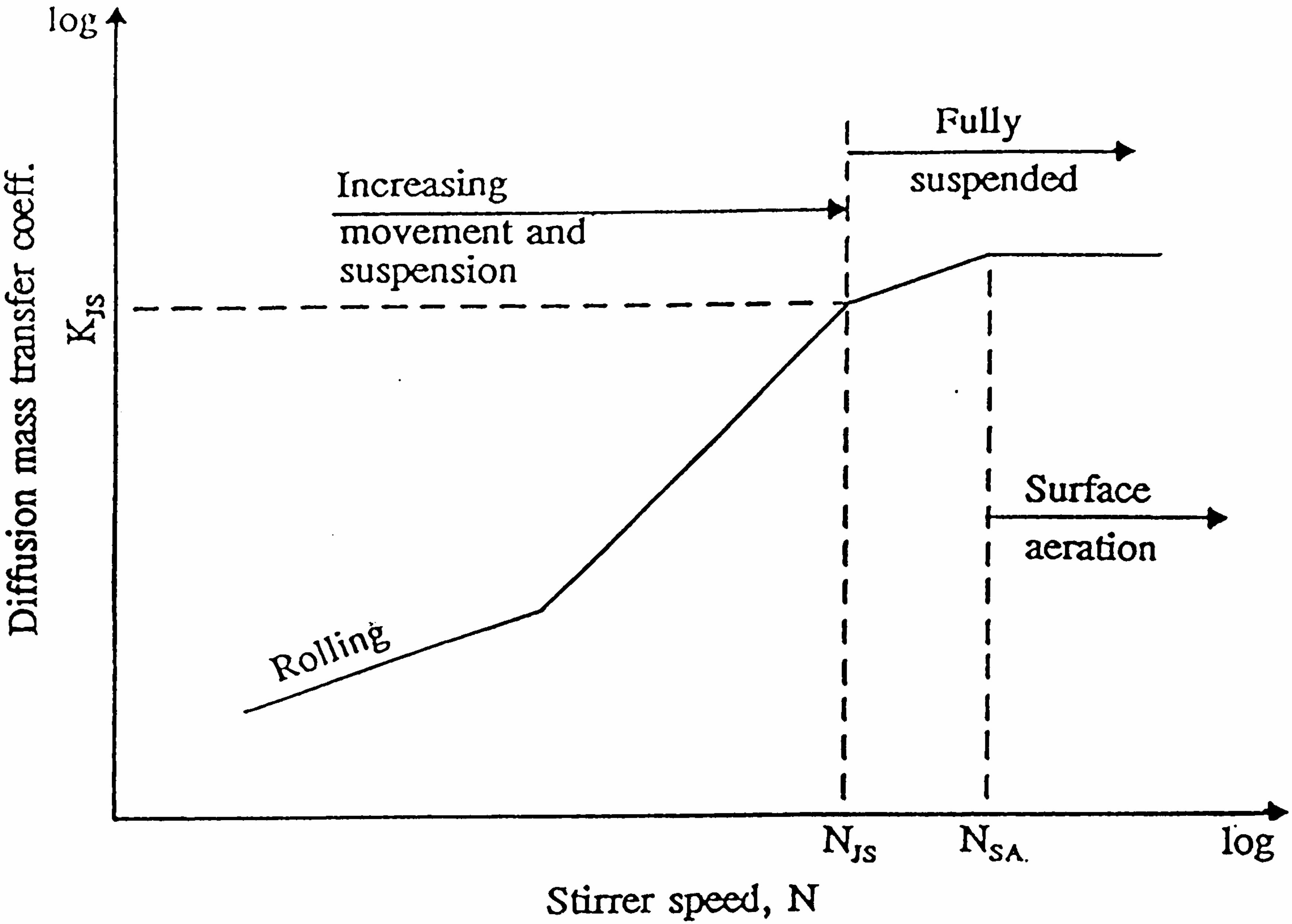


Figure 2. Solid-liquid mass transfer coefficient over a wide range of stirrer speeds.

Solids Suspension and Mass Transfer

- A great deal of work has been done in this area (see Davies, Nienow, Armenante & others).

- Data are correlated by Sherwood No.

$$Sh = \frac{k_L d}{D_{AB}} = 2.0 + xRe^{y} Sc^{1/3}$$

- Values of *x* and *y* appear to be dependent on particle size.

- Need to compare particle size with eddy length scale in defining Reynolds No.

Settling Rate

- Laminar - Stoke's Regime:

$$U_{TO} = \frac{(\rho_S - \rho_L) g d^2}{18\mu}$$

$$\frac{U_{TO}^2}{(\Delta\rho / \rho_L) g d} = \frac{1}{18} \frac{\rho_L U_{TO} d}{\mu}$$

$$Fr_G = \frac{Re_P}{18}$$

Settling Rate

- Turbulent - Newton's Regime:

$$U_{TO} = 1.74\left(\frac{(\rho_S - \rho_L)gd}{\rho_L}\right)^{1/2}$$

$$\frac{U_{TO}^2}{(\Delta\rho/\rho_L)gd} = 1.74^2$$

$$Fr_G = 3.0$$

Settling Rate

- Transitional - Allen's Regime:

$$U_{TO} = 1.04\left(g\,\frac{(\rho_S - \rho_L)}{\rho_L} \right)^{0.72} \frac{d^{1.18}}{\nu^{0.45}}$$

$$\frac{U_{TO}^{1.44}}{((\Delta\rho/\rho_L)\,gd)^{0.72}} = 1.04\,\frac{U_{TO}^{0.44} d^{0.46}}{\nu^{0.45}}$$

$$Fr_G = 1.04 Re_P^{0.3125}$$

SOLID SUSPENSION

- What is needed?
- OFF BOTTOM
- Dissolving and many chemical reactions
  - Off bottom to maximize surface area
- Some storage and delivery
- UNIFORM DISTRIBUTION
- Sampling
- Crystallization and precipitation
- Feeding

2.1 Mechanistic models for solid suspension

- **Turbulence model**

  Suspension due to energy transfer from turbulent eddies of size similar to that of d_p .

- **Fluid velocity model**

  Suspension due to average velocity and hydrodynamic forces (lift and drag) acting upon the particles.

Which is correct?

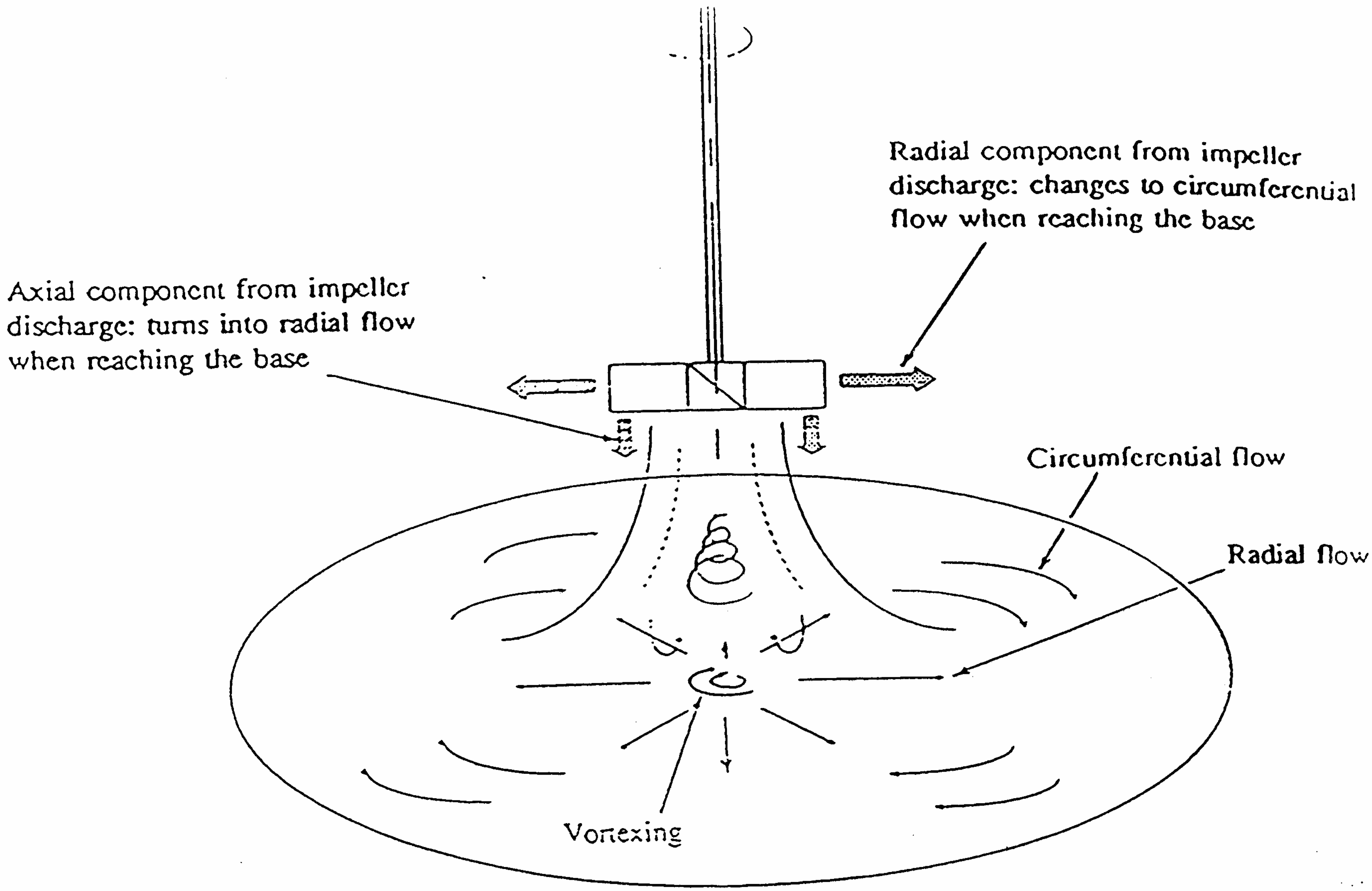


Figure 4. Relationship between the flow patterns discharged by an impeller and those acting on particles on the vessel base.

2.1 Models for solid suspension (ct.)

Neither of the two classes of models alone represent the exact physical conditions.

Both convection and turbulence act on settled particles.

2.2 Semi-empirical correlation by Zwietering

$$N_{JS} = s\,\nu^{0.1}\left(\frac{g\Delta\rho}{\rho_L}\right)^{0.45} X^{0.13}\, dp^{0.2}\, D^{-0.85}$$

No solids remain at the vessel base for more than 1-to-2 seconds

Extensive data sets can be correlated

Some theoretical justification

Limitations and applicability

- "s" values have to be known
  - Characteristic of geometry only - maybe

- not applicable outside the experimental conditions

- applies to free and hindered settling conditions (i.e. low to moderate X), X is weight of solids divided by weight of liquid times 100 - UGH

  but may not be valid or relevant in significantly non-Newtonian systems (i.e. at high X)

Table 1. Geometrical constant, s, values for three different impeller types in a flat base vessel.

s values at C/T =		**1/3**	**1/4**	**1/6**	**1/8**
RDT	D = T/2	4.72	4.2	3.67	3.41
	D = T/3	8.37	7.43	6.50	6.03
PBT4-45	D = T/2	4.97	4.74	4.52	4.40
	D = T/3	6.95	6.64	6.32	6.16
HE-3	D = T/2	7.14	6.82	6.50	6.35
	D = T/3	10.41	9.95	9.48	9.25

Table 2. Operating conditions over which Zwietering's correlation is obtained

Parameter	Range covered
$\Delta\rho$ (kg/m^3)	560 – 1800
d_p (μm)	140 – 520
X (%)	0.5 – 20
T (m)	0.1 – 0.6
D/T	1/6 – 1/2
n_b	4
C/T	1/20 – 1/2
Impel. type	4
Vessel base shape	Flat, dish & conical
μ_L (mPa s)	0.3 - 9

Z CORRELATION

- Since has been extended to many more geometries and sizes and densities

- Not perfect but conservative and nothing better

Main Findings

- **Impeller Geometry**
  - **Axial flow impellers (e.g. hydrofoils) pumping downwards are the most; radial flow (e.g. 6DT) and saw tooth impellers are the least energy efficient for off-bottom suspension.**
  - **Low C generally more energy efficient, except at extremes (e.g. D/T>0.5 C/T<1/6)**

Findings

- **Tank Geometry**
  - **Flat base tanks and dished**
  - **not recommended: cone**
  - **Multiple impellers for processes where H varies**

2.3 Scale-Up Criteria for Suspension Speed

- **Geometrical similarity (Figure 5)**

$$\frac{D_1}{T_1} = \frac{D_2}{T_2}; \frac{W_1}{D_1} = \frac{W_2}{D_2}; \frac{H_1}{T_1} = \frac{H_2}{T_2}; etc.$$

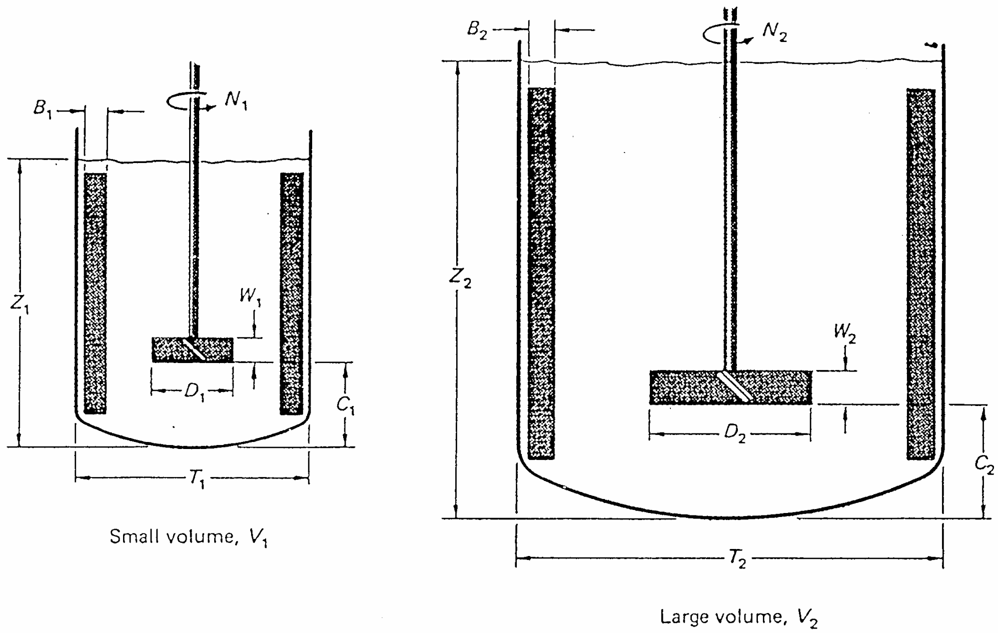


Figure 5. Geometrical similarity as an important scaling criteria

2.3 Scale up (ct'ed)

- **Zwietering's Correlation**

$$N_{JS} \propto D^{-0.85}$$

N_{JS} decreases as scale increases, however, there is some disagreement between workers on the exact value of the exponent on *D*.

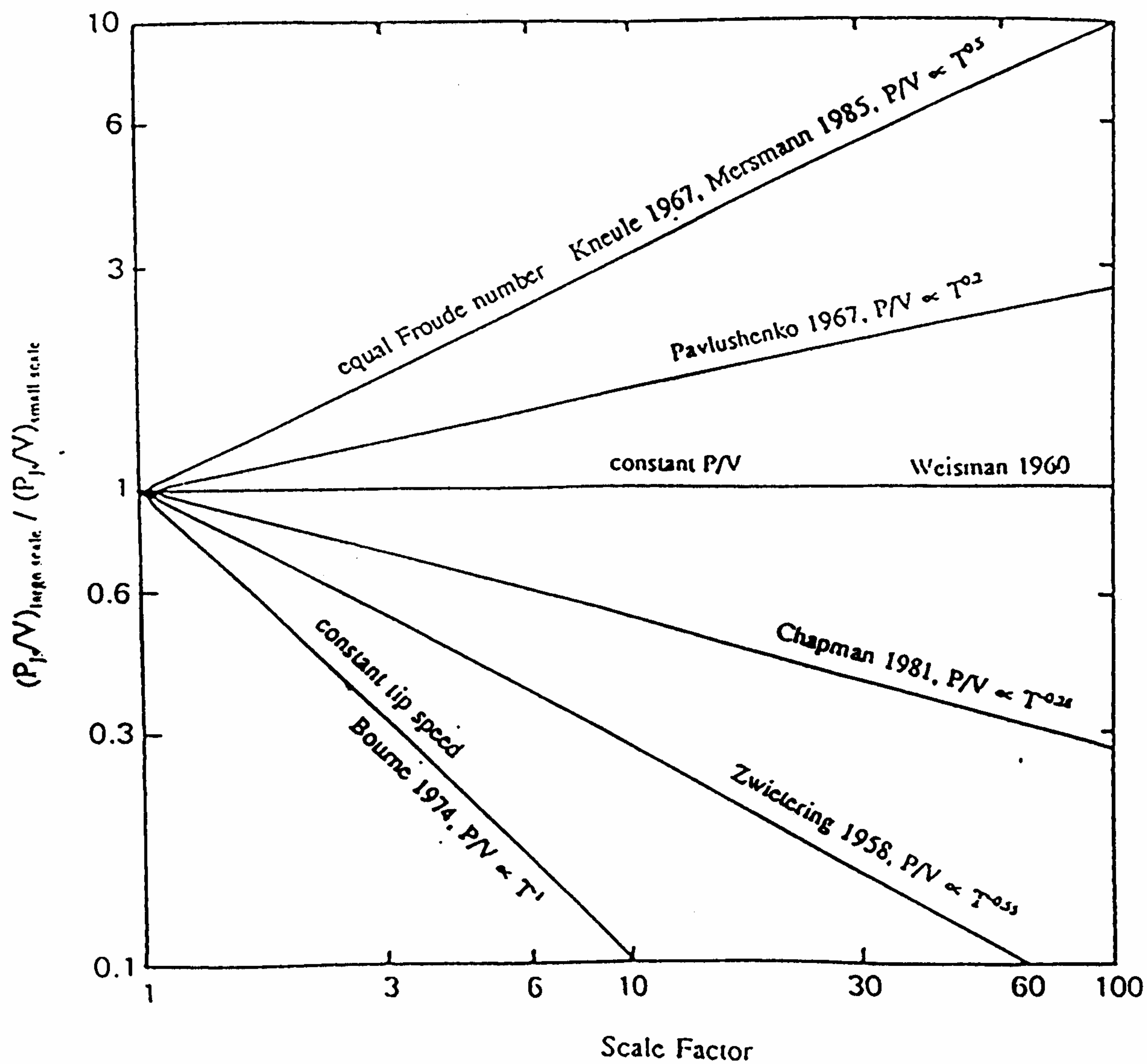


Figure 3. Scale-up rules for solid suspension as published in literature.

SETTLING RATE AND Z

- Zwietering suggests

- Njs proportional to density difference to 0.45 and particle size to 0.2
- Settling rate is density difference times particle size square or to the first
- Density is much more important
- Most data in transitional settling rate

SETTLING RATE AND Z

- At extremes – very large and very small particles – Z breaks down
- Is there another group missing? –
  - Galileo or Archimedes Number
  - Separates settling regions
- Settling rate is in quiet liquid
  - What is the effect of settling in turbulent field?
- Stay tuned

SUMMARY

- Zwietering is for mainly sand of several 100s microns in water – good simulant
- Use Zwietering directly or from scale down experiments
- Note – Zwietering is NOT settling rate
  - Density dominates not particle size

3. SOLID DISTRIBUTION - Introduction

Off-bottom suspension ⇒ mass transfer

Certain degree of homogeneity required for other processes

The mechanisms considered for distribution: <u>convection</u> and <u>turbulence</u>. Convection distributes particles by drag force, the turbulence eddies act to distribute at a smaller scale by fluctuating drag forces acting in all directions.

3.2 Measurement and assesment

- Different experimental techniques: conductivity, sampling, slurry height,…

- Mixture quality assessed in terms of:
  - slurry height,
  - Relative Standard Deviation,
  - concentration profile

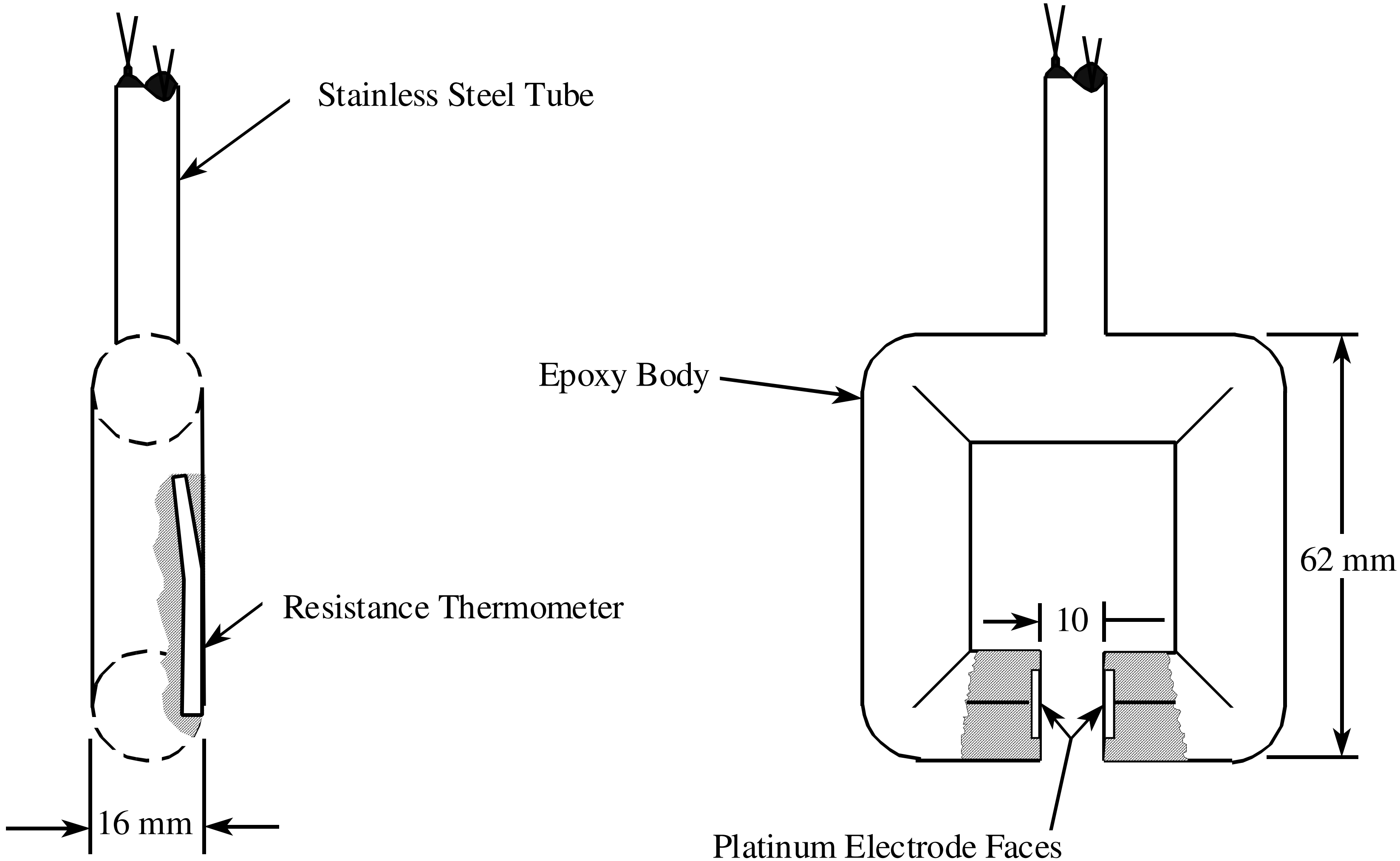


Figure 6. Conductivity probe used at BHR Group to measure local solids concentration

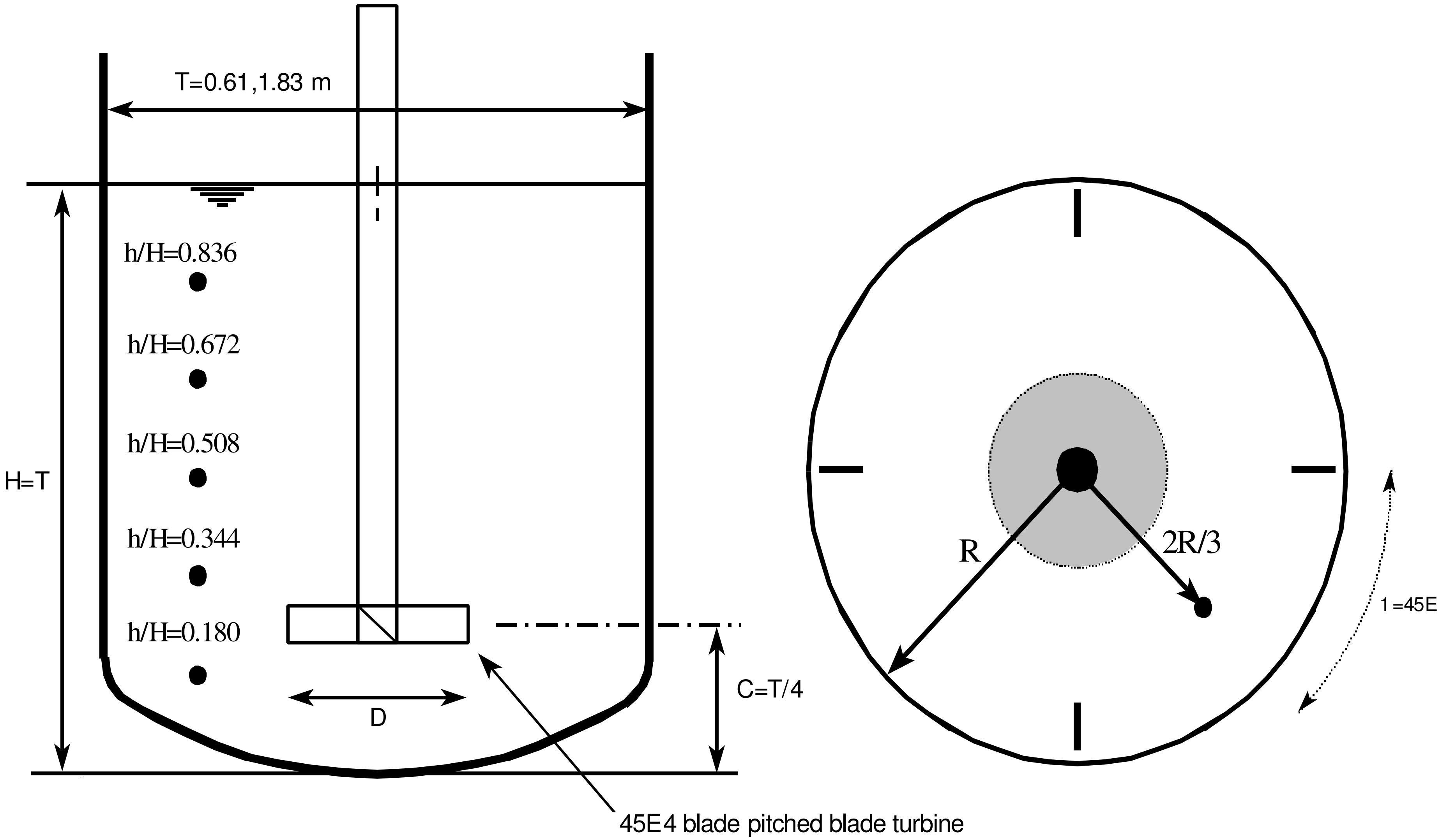
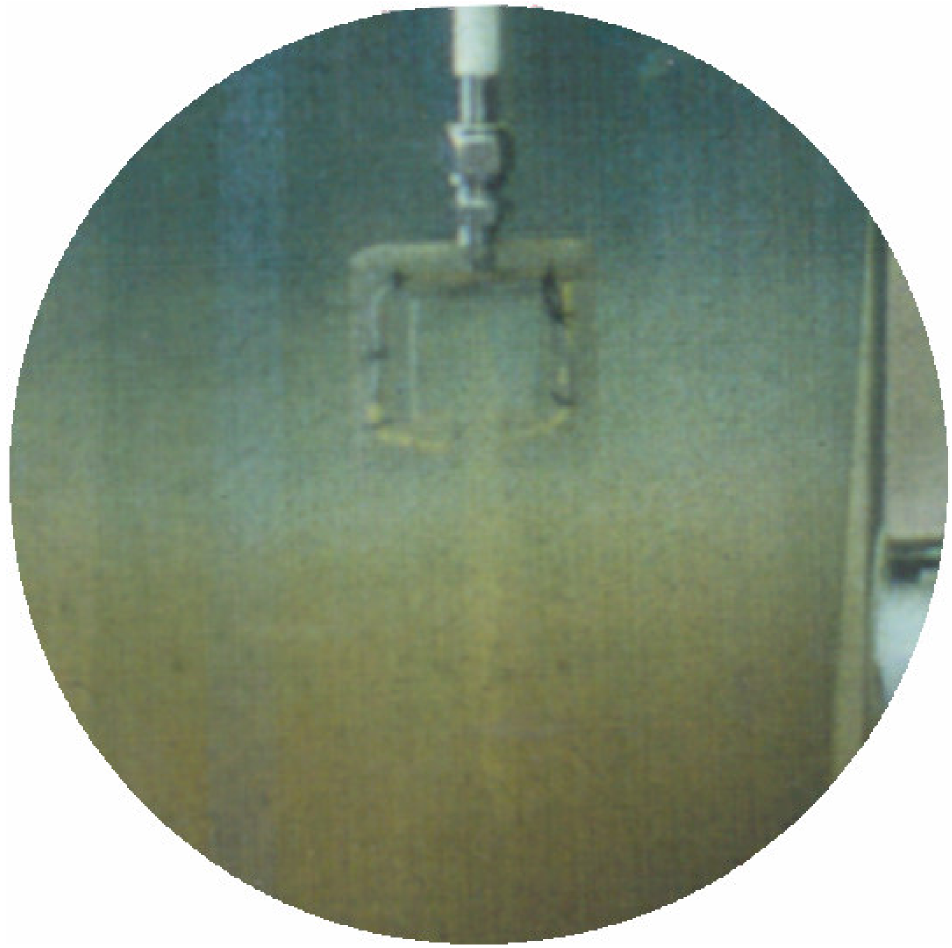


Figure 7. Measurement positions in experimental studies at BHR Group (Taylor, 2000)


beam



Relative Standard Deviation (RSD)

$$RSD = \frac{1}{C_m}\left[\frac{1}{n-1}\sum_{1}^{n}\left(C_{ij} - C_m\right)^2\right]^{\frac{1}{2}}$$

C_m: Calculated mean bulk solid concentration
C_{ij} : Measured local solid concentration
n : Number of samples

RSD = 0 for perfect homogeneity

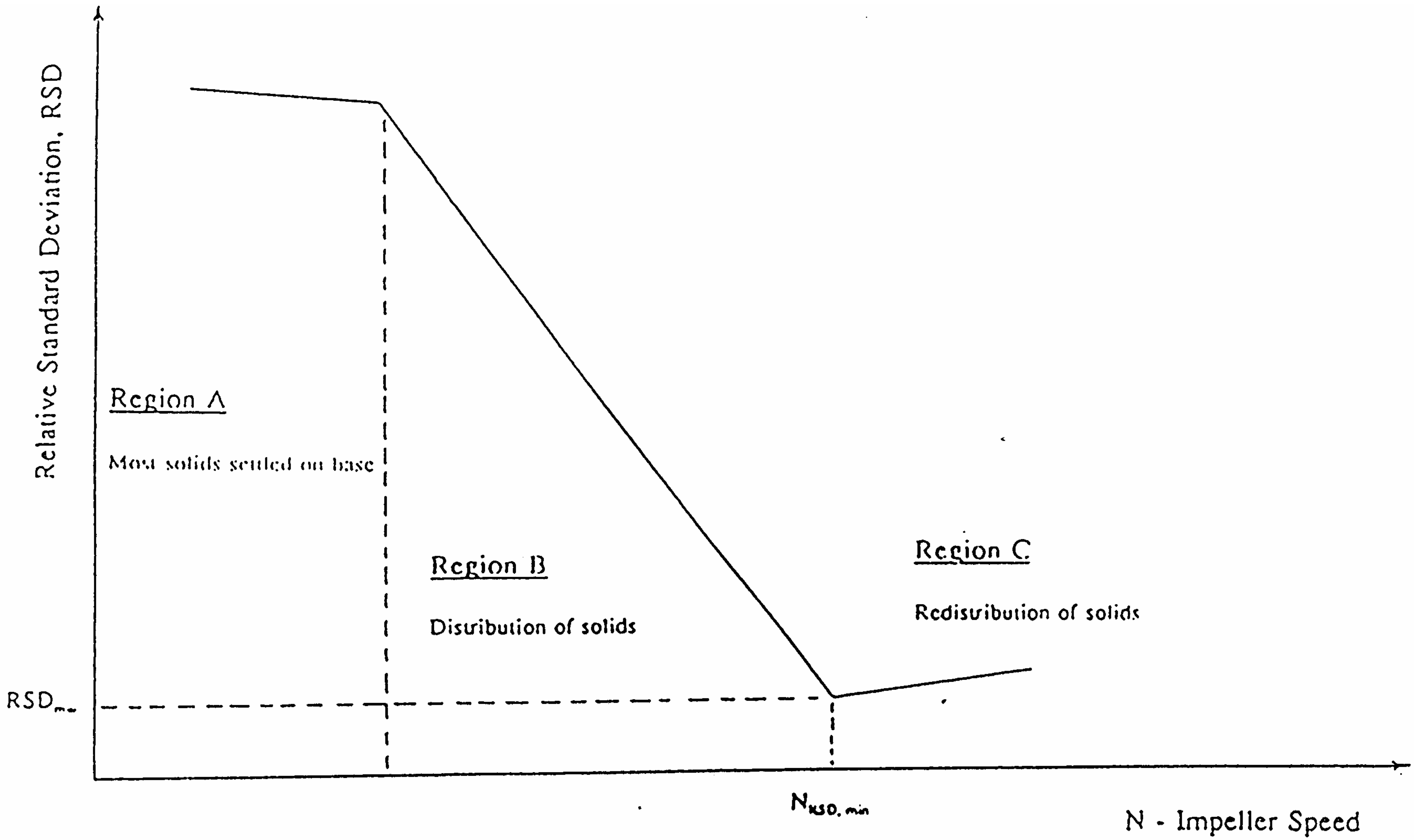


Figure 9. A typical solid distribution curve.

3.2. Measurement and assessment (ct'ed)

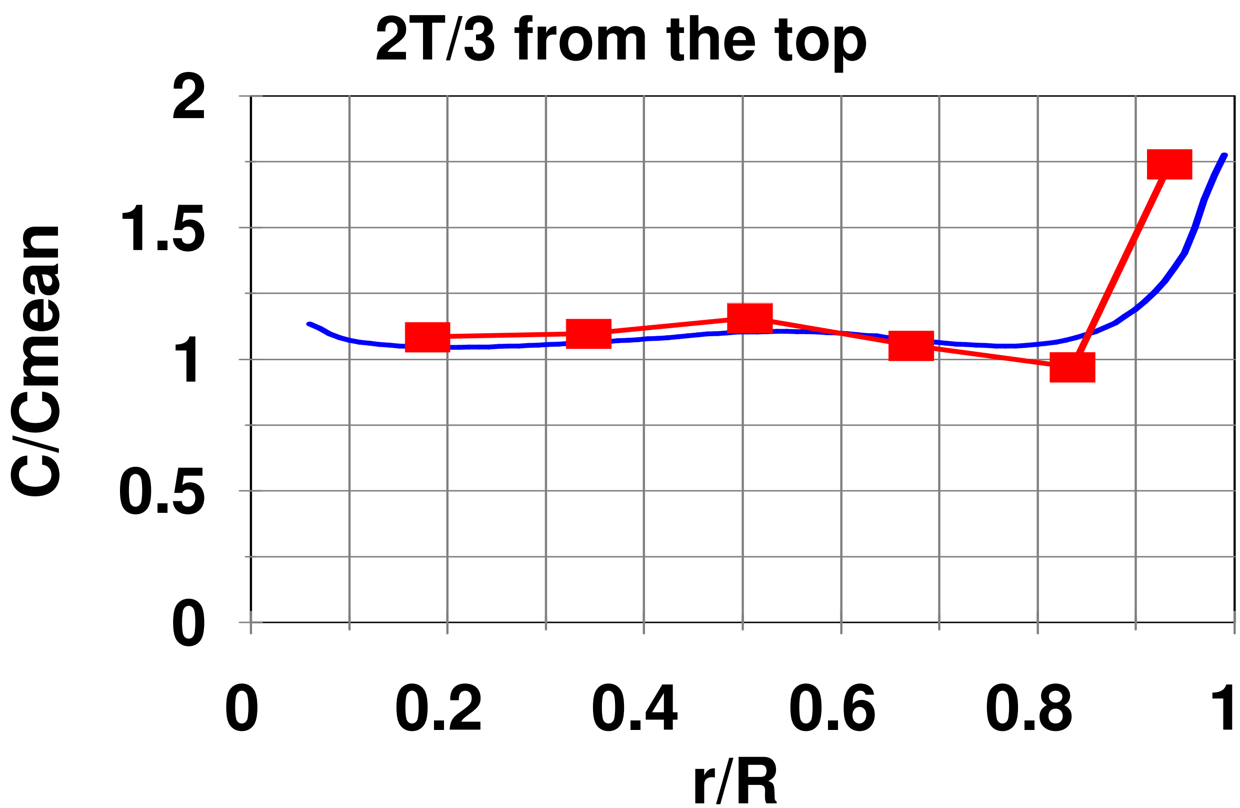
- RSD a single value assigned to the whole mixture

  but does not provide information on how solids are distributed

- Concentration profiles required for a detailed assessment and

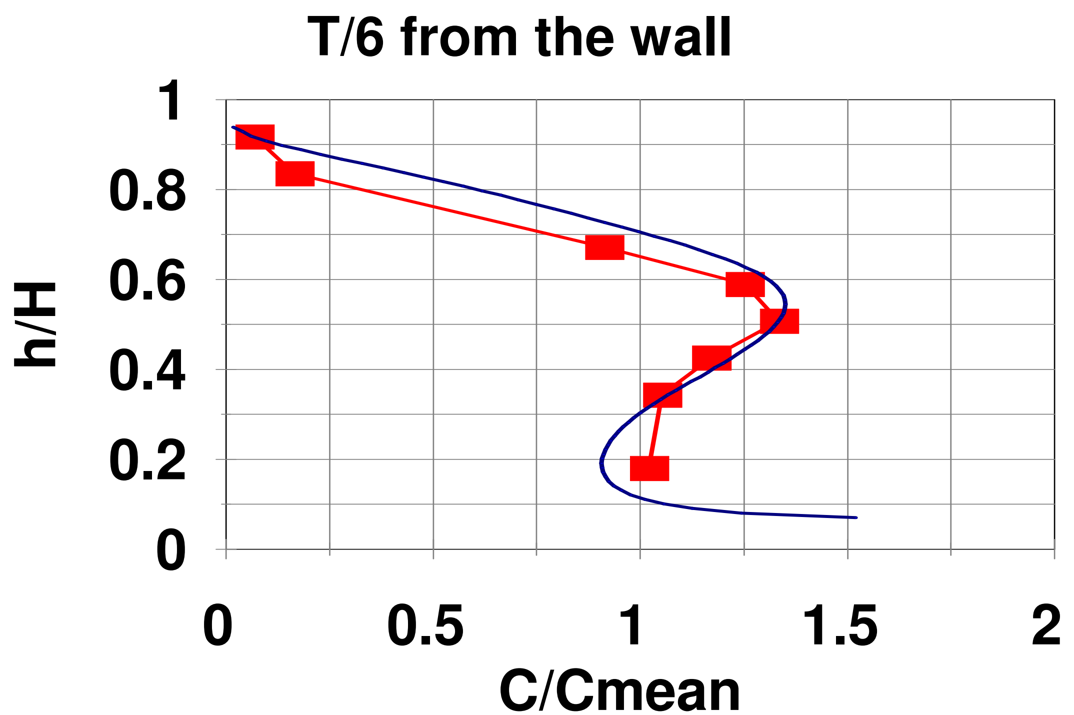
  and to correctly position the inlet and outlet in a CSTR

3.3 Major points related to solids



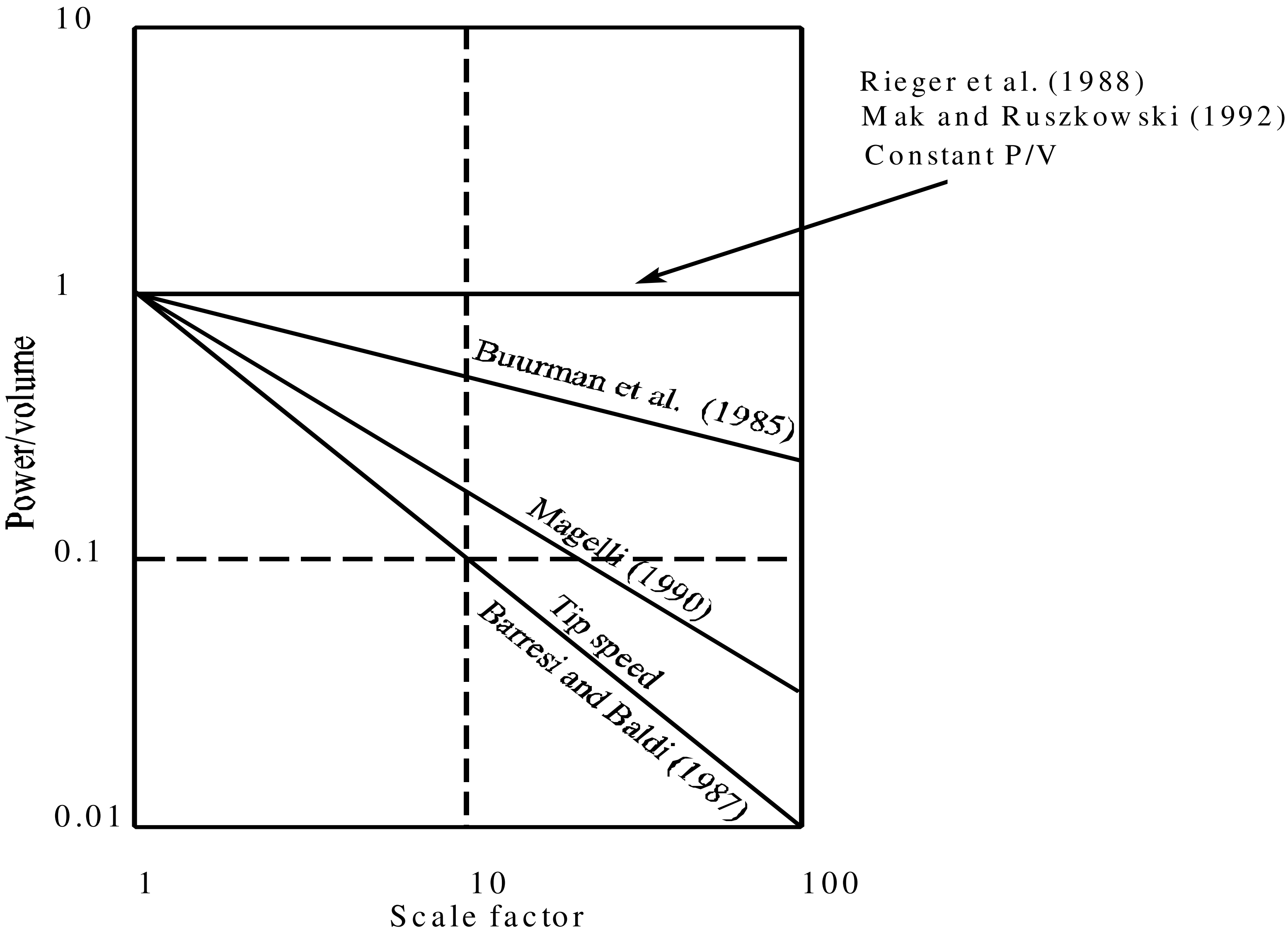
Practically uniform radial concentration profiles

3.3 Major points on solids



- **"Belly plot" on the axial traverse**
- **Position and value of max. depends on *N, C/T, D/T*, particle properties,...**

3.3 Major points on solids



4. DUAL IMPELLER CONFIGURATIONS

- Commonly used in ‘tall’ tanks
- No advantage for suspension: P_{js} higher than in a single impeller configuration.
- Dual impellers markedly improve distribution quality

5. SOLID SUSPENSION USING A JET

- alternative to mechanical agitation
- good off bottom suspension but poor top-to-bottom distribution
- limited access above the vessel, there may not be enough headroom to install a gear box and motor
- portable/flexible

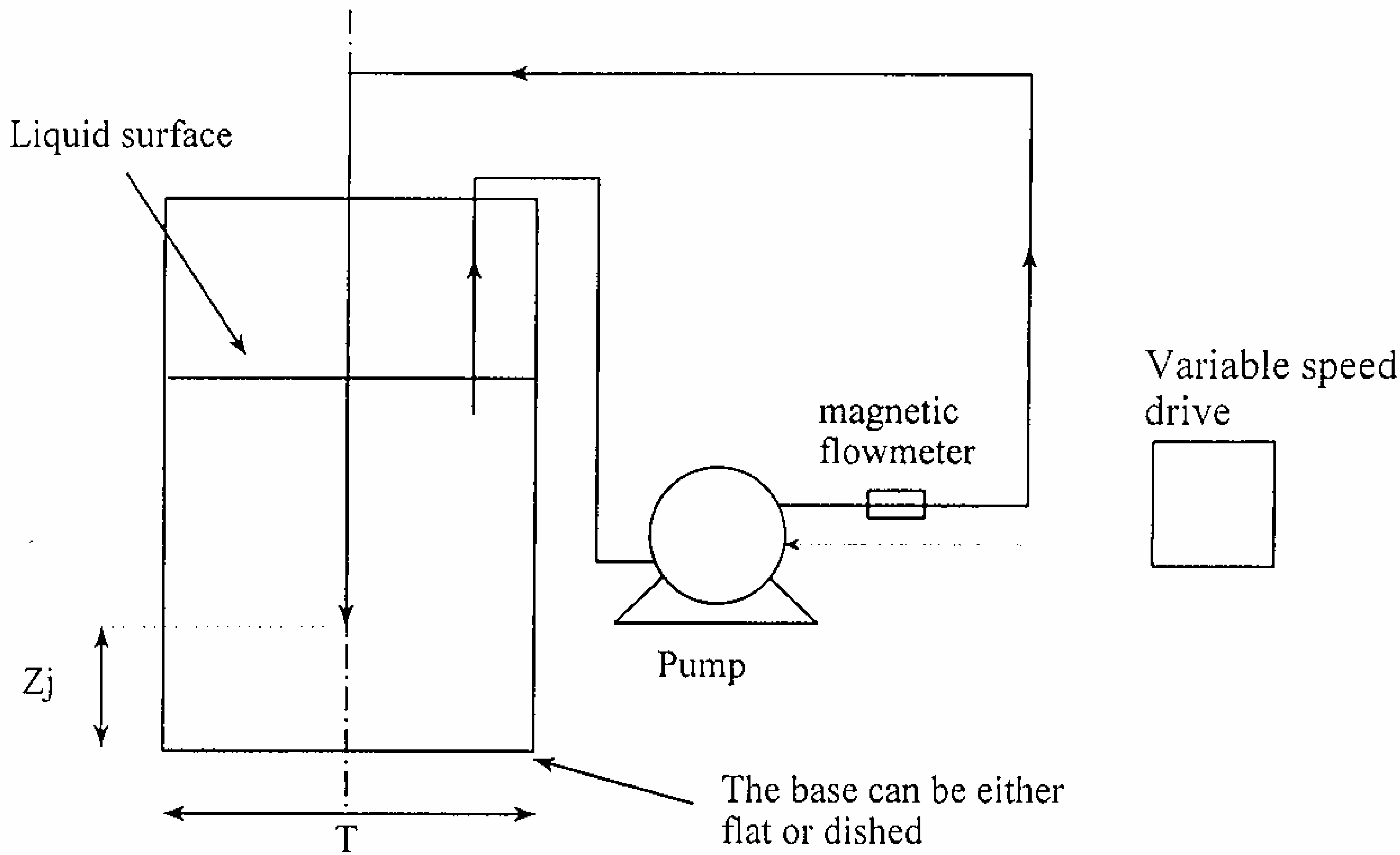


Figure 16. A typical arrangement of a jet mixer for solid suspension.

Advantages of using jets

- mechanical constraints on agitator size in very large vessel

- energy efficiency for solid suspension in comparison to agitation tanks depends on the specific design

- easier to install in wide, shallow vessels: agitator system requires extra supports

Advantages of using jets

- No moving parts in vessel : jet motion induced by an external pump (reduced maintenance)
- low maintenance cost: jet mixing system is very simple and pump is remote from the tank
- low capital cost: pump may already be present for tank drainage.

PULSE JETS

- Not steady
- Multiple jets in one tank
- Being studied for waste work
  - Parsons/SWPF, Battelle/WTP
- Off bottom similar to steady if short off time or slow settling
- Distribution strongly affected

Effect of Physical Properties

- The effects of changes in settling rate from changes in physical properties can be predicted:

$$d \uparrow \Rightarrow U_{TO} \uparrow$$

$$\frac{\Delta\rho}{\rho_L} \uparrow \Rightarrow U_{TO} \uparrow$$

$$\mu \uparrow \Rightarrow U_{TO} \downarrow$$

- Effect of concentration:
  – Increasing concentration slows settling velocity.

Mechanistic Approach

- Solids suspension is not governed by balance between settling velocity and mean flow.
- Role of turbulent eddies?
- Pick up and re-suspend particles from the vessel base.
- Baldi (*Chem Eng Sci.* 1978) looked at the gravitational force acting on a particle and the fluid force acting on it due to turbulence.

$$(\rho_S - \rho_L) g d^3 = K \rho_L (u')^2 l_E^2$$

Mechanistic Approach

- The size of the eddies influencing the motion of the particles will be the size of the particles

$$l_E \approx d \qquad\qquad u' \propto \sqrt{gd(S-1)}$$

- The fluctuating velocity can be related to the local energy dissipation rate:

$$u' \propto (\varepsilon l_E)^{1/3} \rightarrow (\varepsilon d)^{1/3}$$

- The local EDR is proportional to the average power input per unit mass:

$$\varepsilon \propto Po N_{JS}^3 D^2 \left(\frac{D}{T}\right)^3$$

- N_{JS} is the impeller speed required to just suspend the particles.

Mechanistic Approach

- Re-arranging: $$Po^{1/3} N_{JS} D^{2/3} \left(\frac{D}{T} \right) d^{1/3} \propto \sqrt{gd(S-1)}$$

$$N_{JS} = s \frac{d^{1/6} (g(S-1))^{1/2}}{Po^{1/3} D^{2/3}} \left(\frac{T}{D} \right)$$

- *s* is a dimensionless constant. Its value must be determined experimentally.
- Note: weak effect of particle size and no effect of viscosity.

Zweitering - Experimental

- Work done in late 50's.
- Measured speed required to just suspend particles.
- No particle stationary on vessel base for longer than 1 - 2 seconds.

$$N_{JS} = s\frac{\nu^{0.1} d^{0.2} X^{0.13} (g(S-1))^{0.45}}{D^{0.85}}$$

$$X = 100 \times \frac{M_S}{M_L}$$

- Correlation confirmed in vessels up to 9 ft. diameter.
- Very similar functionality to mechanistic form.

Values of “s”

Impeller	*D* / *T*	*c* / *T*	*Po*	*s*
Hydrofoil	0.33	0.25	0.30	9.3
Hydrofoil	0.50		0.30	7.0
PBT – 45	0.33		1.73	4.6
PBT – 45	0.50		1.53	6.1
FBT – 90	0.33		3.19	4.4

- Hydrofoils need to operate at higher speeds than PBT’s.
- Which impeller type is most energy efficient for solids suspension?

Scale-Up

- Very confident about Zweitering's correlation:
  - Good set of data.
  - Taken at large scale.

- What happens to power requirements on scale-up?

$$\frac{N_L}{N_S} = \left(\frac{D_S}{D_L}\right)^{0.85} \qquad \frac{\varepsilon_L}{\varepsilon_S} = \frac{N_L^3 D_L^2}{N_S^3 D_S^2}$$

$$\frac{\varepsilon_L}{\varepsilon_S} = \left(\frac{D_S}{D_L}\right)^{3\times 0.85-2} = \left(\frac{D_S}{D_L}\right)^{0.55}$$

- Power input per unit mass decreases on scale-up!

Buurman -5th Europ Conf. On Mixing

- Buurman worked for Shell in the Netherlands.
- Studied particle distribution in vessels up to 63 m^3:
  - Solids suspended to 90 % of liquid depth.
- For $H = T$, $D = 0.4\ T$ and $L = 0.9\ T$:

$$\frac{\rho N^2 D^2}{g \Delta\rho d}\left(\frac{d}{D}\right)^{0.45} \geq 20$$

- For fixed geometry and physical properties:

$$N^2 D^{1.55} = K \qquad\qquad N^3 D^{2.33} = K'$$

- Almost constant power input per unit mass.

Mak / FMP Consortium

- FMP Consortium has published early work on solids distribution.
- Measured distribution in vessels of 2, 6 and 9 feet diameter.
- Used conductivity technique:
  - Conductivity proportional to slurry concentration in probe volume.
  - Calibrated in fluidized bed.
- Measured concentration at different axial positions.
- Best work in field.

Data Analysis

- Defined: Relative Standard Deviation of Concentration:

$$RSD = \frac{1}{\overline{C}} \sqrt{\frac{\sum_{i=1}^{i=n} (C_i - \overline{C})^2}{n-1}}$$

- Plot *RSD* versus mixing parameters (speed, power input, Froude No. etc. etc.).
- Look for parameter that "collapses" curves taken at each scale.
- Look for relationship between distribution and N_{JS}.

Distribution versus N_{JS}

- Some impellers achieve uniform dispersion when operating at N_{JS}, e.g. large PBT.
- Others have poor distribution, e.g. hydrofoils.
- Operation at "just suspended" point is no guarantee of uniform dispersion.
- An area of active research
  - CFD and BHRG FMP experiments

Example of C_i versus N

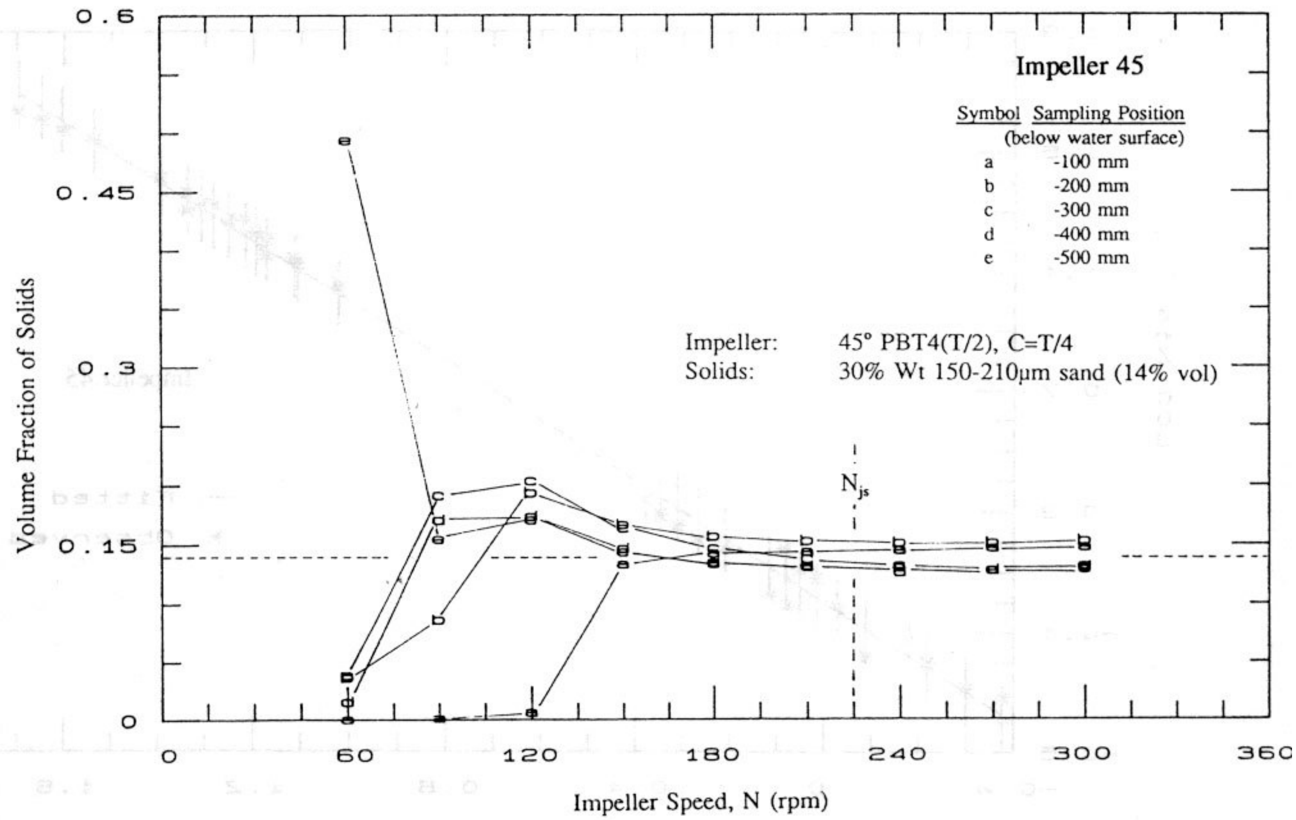


Fig 13 Plot of Solids Concentration against Speed at different Sampling Locations

RSD versus N

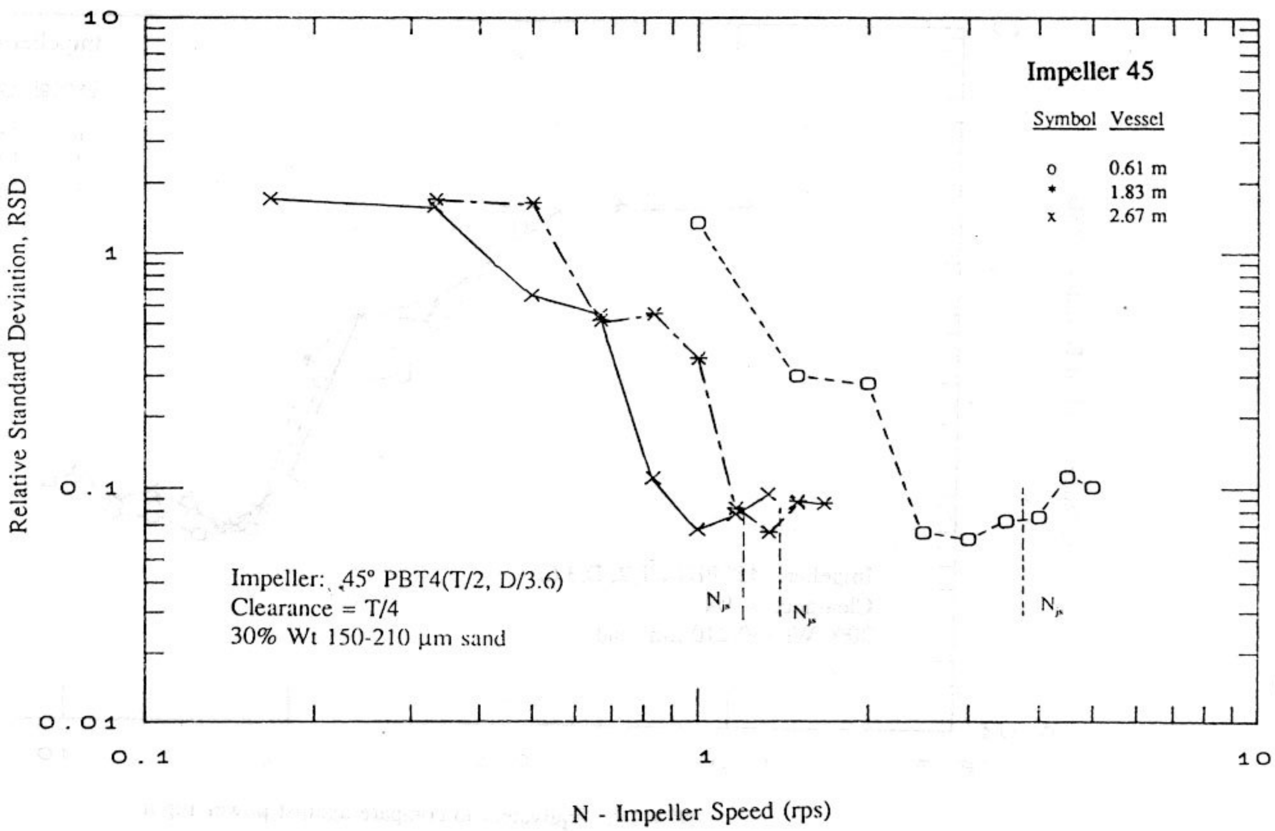


Fig 15 **Plot of RSD against Impeller Speed for 3 Scales**

RSD versus Po N^3 D^2

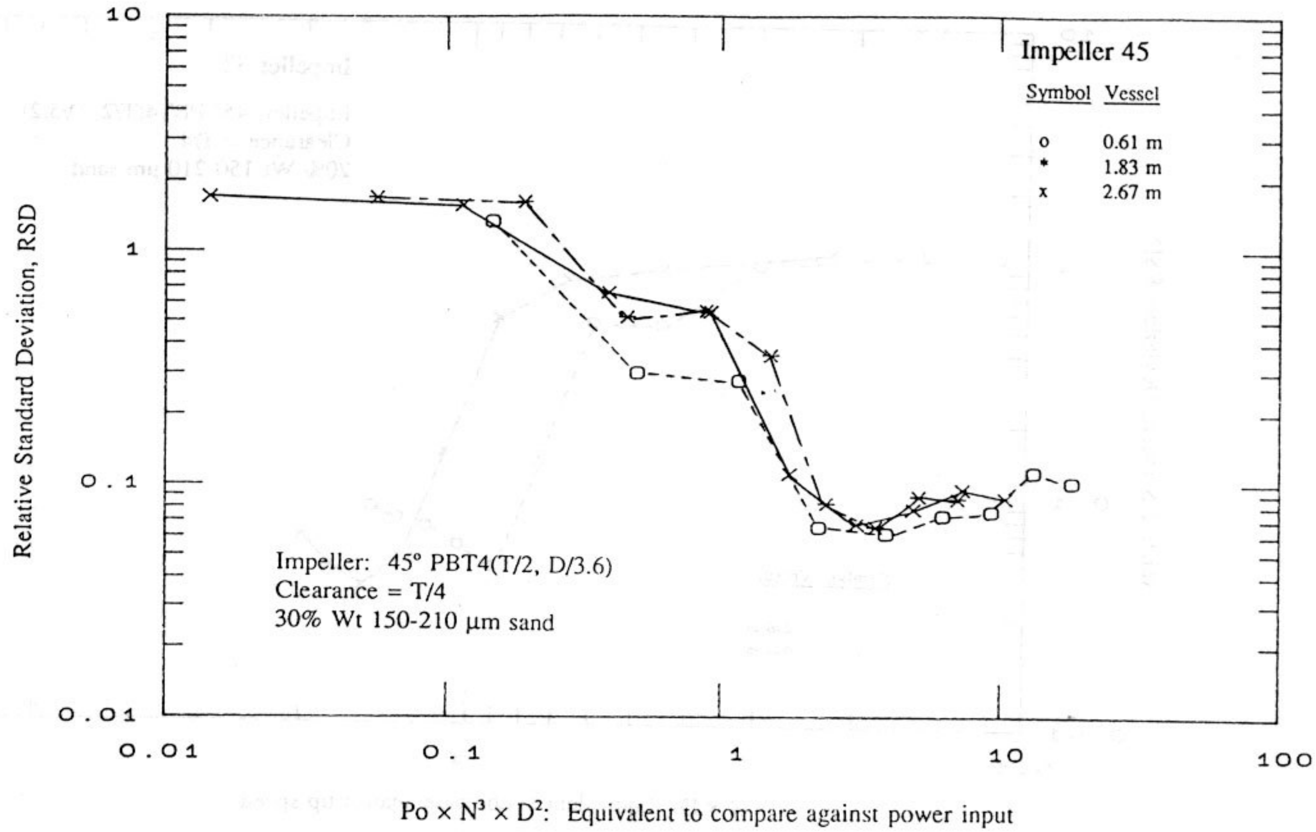


Fig 18 Solids Distribution Scale-up Data: comparing against power input

RSD versus N $D^{0.78}$ - Buurman's Model

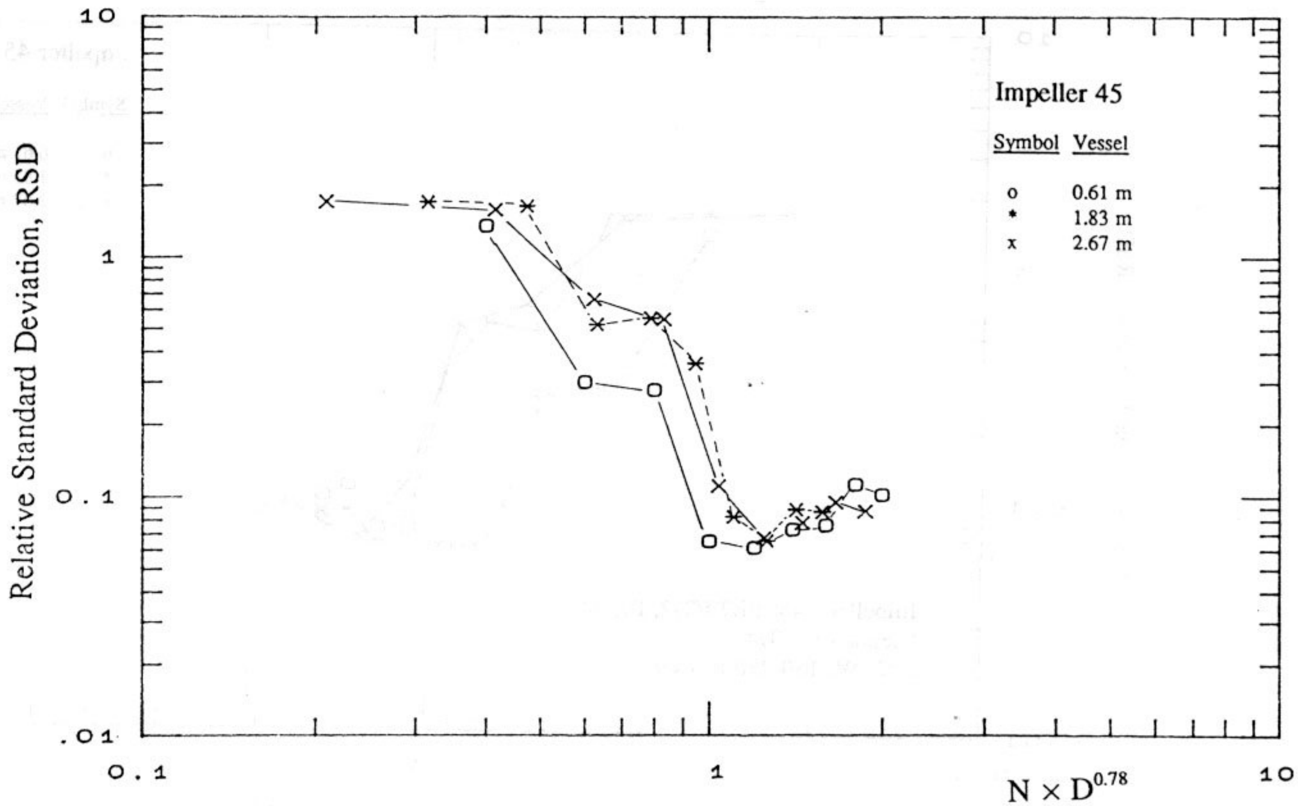


Fig 14 Solids Distribution Scale-up Data according to Buurman's Model ($N_{RSD} \propto D^{-0.78}$)

Plots of RSD - Summary

- *RSD* versus *N*:
  - N_{JS} decreases on scale-up.
  - No relationship exists for *RSD.*

- *RSD* versus $Po\ N^3\ D^2$:
  - Equal power input per unit mass.
  - Very good relationship between *RSD* at each scale.

- *RSD* versus $N\ D^{0.78}$:
  - Buurman's Froude No. model.
  - Good relationship, but not as good as equal power input per unit mass.
  - Seems to work well at larger scale.

Density

- Need to know fluid density for power calculations.

- When $N < N_{JS}$: $\rho_{SL} = \dfrac{1}{\dfrac{x}{\rho_S} + \dfrac{1-x}{\rho_L}}$ $\rho = \rho_L$.

- When $N \geq N_{JS}$: $\rho = \rho_{SL}$.

$$x = \frac{M_S}{M_{SL}}$$

Tickler Turbine

- Many vessels operate on a cycle - filling and emptying.

- If vessel contains slurry, low level impeller must be installed:
  - If not, particles will settle out once impeller is uncovered.

- Typical design:
  - Flat blade turbine: $0.33 > D / T > 0.25$
  - Clearance: Within 6 - 12 inches of vessel base.
  - PBT or Hydrofoil: $D / T = 0.50$
  - Clearance: Normal position.
  - Calculate N_{JS} for main impeller.

- Add power of each impeller.

ATTRITION / COMMUNITION

- Reducing particle sizes
  - May be good or bad
- Many particles are agglomerates of primary particles
- Many crystals are fragile
- Size reduction correlates with power per unit volume and time or specific energy
  - Batch and continuous

ATTRITION

- Dupont work
- Used agglomerates – carbon beads
- Breakage by fines coming off agglomerates
  - Common mechanism
- Rate process with time depending on local energy dissipation
- Similar findings on communition in media mills.

ATTRITION

- Do not reach equilibrium quickly
- Time times power
- Hours in a tank can be equivalent to one pass through a pump
- Linear relation

ATTRITION

- Fine particles caused by attrition often limit separation processes
  - Filtration and centrifugation
- Reduce attrition by de-energizing processes
  - Reduce energy input
  - Reduce contact time
    - Holdup in storage tanks
    - recycle

End of M7