

# AIChE Webinar: Solid-Liquid Separations: Fundamentals and Applications

Overview: Karl Jacob  
Principles and Practices: Ray Collins

# Solids Processing Webinar Series

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3. Introduction to Pneumatic Conveying of Solids 9/8/10
4. Solid-Gas Separation: Fundamentals and Applications 1/19/11
5. Introduction to Fluidized Bed Technology 4/13/11 (with Ray Cocco)
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8. Mixing and Blending of Solids, 10/30/13
9. **Solid-Liquid Separation, today (with Ray Collins)**
10. Population Balance Modeling for Solids Processing 5/14/14 (with Ben Freireich)
11. Size Reduction and Grinding of Solids 9/10/14
12. More to come...

# Overview of Solid/Liquid Separation

- Few people appreciate the breadth to which solid/liquid separation is applied to our lives today
  - Automotive oil filters
  - Filters found in our kitchens
  - Process filters – hundreds of both bag and cartridge filters are found in the chemical plants today
  - Screens used to keep fish out of water intakes
  - Centrifuges to separate solids out of slurries
  - Fine (nano-) particle filtration used in semiconductor processing
  - Settling in waste water treatment
  - And **thousands** of other applications...
- Today we will not address items such as reverse osmosis or gas filtration (see webinar #4 for solid/gas separation)

# Solid/Liquid Separation (SLS)

## Facilitating Cutting Edge Technology Development

- SLS is often seen as a mature technical area where only incremental improvements in technology made – this is not true.
- As bioreactors find increased use in chemical processing, often solid products are only present at one wt% or less – SLS is the key to economical separation of the valued product in this sea of water. The problem is doubly difficult because of the deformable cell bodies.
- As we can now routinely grind solids into the submicron and nanosizes, the use of membrane filtration to make carefully sized separations of these particles can improve product performance.
- With the emphasis on potential shortages of both potable and process water, the demands on SLS will only increase over the coming years as society demands both clean water and a clean environment.

# Solid Liquid Separation Problem Solving

- It must be emphasized that there is no “one size fits all” solution to SLS problems
- Recognize that SLS can be used in conjunction with other solids processing unit operations like drying
- A systems approach is often useful in determining “best” piece of equipment to solve a given problem
  - Particle size and size distribution – rocks or nanoparticles?
  - Particle concentration – dense slurry or isolated contaminant?
  - Nature of the particle collected – product vs. waste stream
  - Batch vs continuous?
  - What are the pieces of equipment both ahead of and after the solid/liquid separation equipment? Careful consideration must be given to optimize the whole system vs. just the SLS equipment
  - New product vs. existing material which you are extremely familiar with

# Separation Mechanisms

- Gravity – in many cases, a static screen or strainer is sufficient to separate larger particles from the liquid. As well gravity settling is used effectively separate solids in waste water treatment plants
- Vacuum or pressure – can be used to essentially suck or press the liquid out of the solids – obvious limitation for vacuum systems so pressure filters – in many cases, cake washing is done in conjunction with vacuum or pressure filtration
- Centrifugal – both hydrocyclones and centrifuges use centrifugal force to enhance the separation of particle from the liquid – numerous formats for centrifuges are available
- Consequence of the above is that there is a significant number of unique pieces of equipment out on the market today and the choices are often bewildering. Let's look at how we might use fundamentals to guide our decision making.

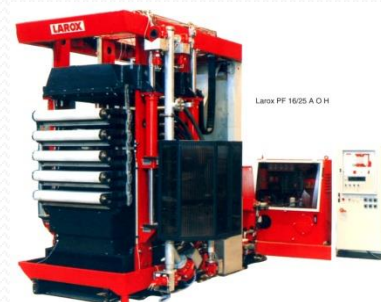
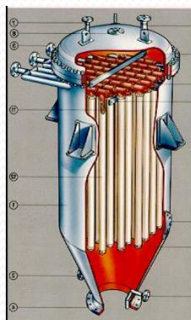
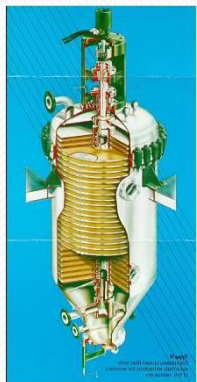
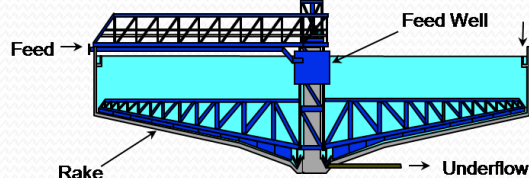
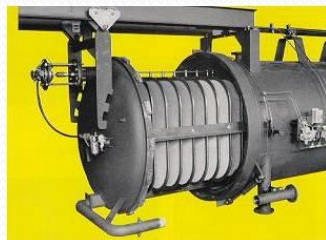
# Perspective on Solid-Liquid Separation Technology

J.R. Couper, W.R. Penney, J.R. Fair, and S.M. Walas wrote in Chemical Process Equipment: Selection and Design, 2<sup>nd</sup> edition published in 2005:

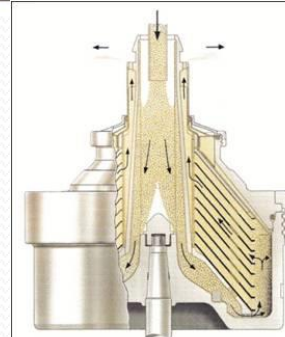
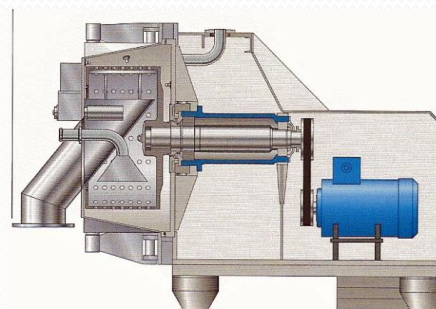
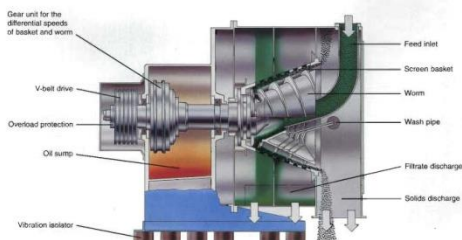
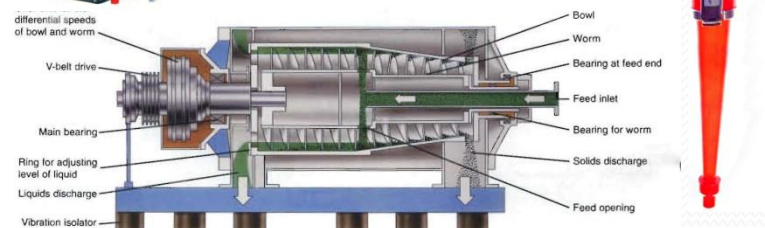
*Solid-liquid is not usually considered a “high-tech” operation. Much equipment for the separation of liquids and finely divided solids was invented independently in a number of industries and is of diverse character. These developments have occurred without benefit of any but the most general theoretical considerations. Even at present, the selection of equipment for specific solid-liquid separation applications is largely a process of scale-up based on direct experimentation with the process material.*



# Usual Emphasis on Equipment



What to Use?





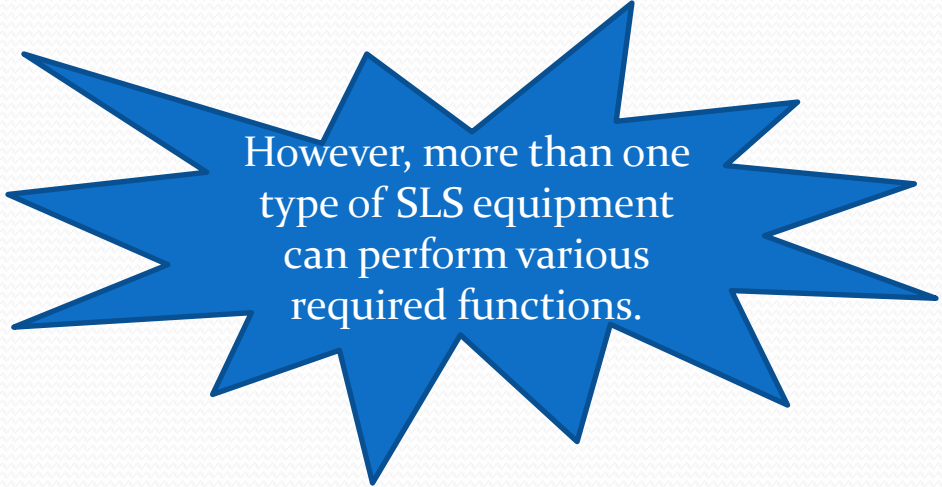
# Classes of Chemical Process Equipment\*

## Proprietary

- Proprietary equipment is designed and manufactured by specialist firms.
- Examples: pumps, compressors, filters, centrifuges, and dryers.
- “Unless employed by one of the specialist equipment manufacturers, the chemical engineer is not normally involved in the detailed design of proprietary equipment. His job will be to select and specify the equipment needed for a particular duty; consulting with the vendors to ensure that the equipment supplied is suitable.”
- The use of standard equipment, whenever possible, will reduce costs.

## Non-Proprietary

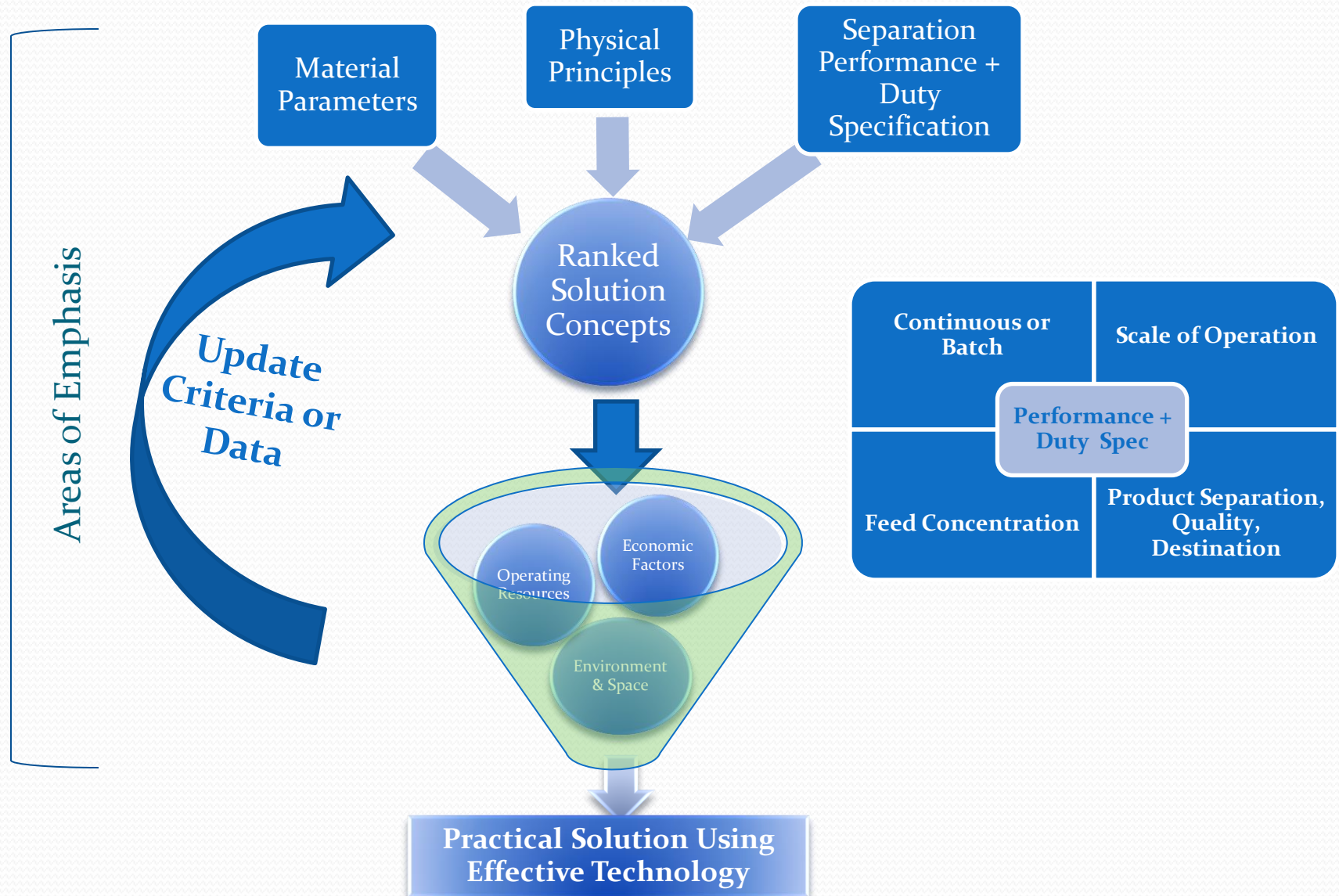
- Non-Proprietary equipment is designed as special, one-off, items for particular processes.
- Examples: reactors, distillation columns, and heat exchangers.



However, more than one type of SLS equipment can perform various required functions.

\*Coulson and Richardson, Chemical Engineering Vol. 6: Design, “Chapter 10: Equipment Selection, Specification and Design”, 4<sup>th</sup> Edition, 2005.

# Elements of Systematic SLS Application Development



# Solid Material Parameters

- **Solids Concentration**

- Gravimetric Measurement of Total Suspended Solids (TSS): Filter/Wash/Dry/Weigh
- Loss-on-Drying (LOD): solutes will contribute to suspended solids wt.

- **Intrinsic (Skeletal) Density**

- Explicit property in the mass and momentum balances leading to design equations.
- Enables conversion between volume and mass thereby yielding concentrations in either basis.
- 10 cc sample conveniently measured using He pycnometer

- **Solid Particle Size Distribution**

- Volume-based (= Mass-based for constant density)
- Number-based (Permeability substantially impacted by minor fines fraction).

- **Particle Shape**

- 3-dimensional shapes tend to form porous deposits of higher permeability.
- Shapes with high aspect ratio (needles, plates, rods) tend to produce lower permeability.
- Can be adequately characterized using Image Analysis of photographs.

- **Deformability under Stress**

- Not measured but important to recognize forms when present:
  - Gels (solid network encapsulating liquid through surface tension effects)
  - Water-swollen bio-solids

## Comparison of Particle Sizing Methods

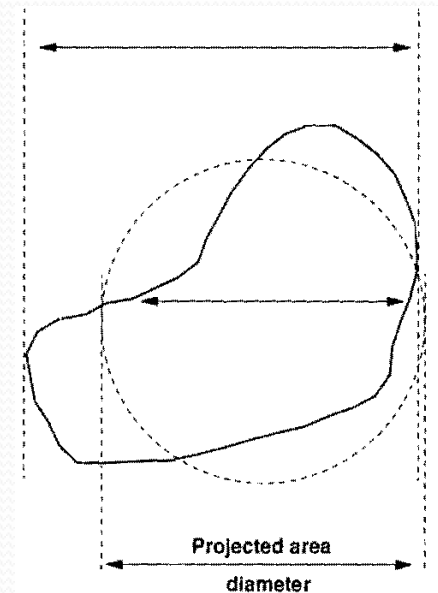
(Solid-Liquid Filtration and Separation Technology, 2<sup>nd</sup> ed., Ruston, Ward, Holdich, Wiley-VCH, 2000)

Means		Technique	Approximate size range, $\mu$	Diameter Measured
Direct	Field scanning	Microscope (add digital camera with image analysis software)	1 - 100	Length, projected area statistical diameters
		Electron microscope	0.001 - 5	
		Scanning electron microscope	0.1 - 5	
		Sieves	45 - 1000+	Mesh size
Indirect	Sedimentation	Andreasen pipette	2 - 60	Stokes' diameter
		Sedigraph		
		Photosedimentometer		Volume
		Centrifuges	0.5-5	Projected area
	Stream scanning	Electro sensing (Coulter®)	1 - 100	Projected area
		Light obscuration (Hiac®)	2 - 150	Volume or projected area
		Light scattering	0.3 - 10	
		Laser diffraction	0.5 - 800	
		Scanning laser microscopy (FBRM)	0.5 - 1000	Random distribution of scanned chords
	Surface Area	Gas adsorption Permeability		Volume-surface or Sauter mean diameter

# Material Parameters: Particle Characterization

- **Specific Surface,  $S_O$** 
  - Ratio of particle surface area to particle volume (or mass)
  - Surface drag is a key factor in filtration
- **Sauter Mean Diameter,  $D_p$** 
  - Diameter of a sphere having the same specific surface as the particle
  - Also known as the volume specific surface or surface volume mean diameter
  - For a spherical particle,  $S_O = 6 / \text{Sauter mean diameter } D_p$
  - Most relevant to filtration mechanism where drag and thus surface area dominates
- **Stokes Diameter,  $d_{st}$** 
  - Diameter of a sphere having the same density and velocity as the particle, settling under laminar flow in a fluid of the same density and viscosity
  - Most relevant to sedimentation
- **Equivalent Sphere Diameter,  $d_{es}$** 
  - Diameter of sphere having projected area equal to that of particle.
  - Used by several techniques of particle size measurement.
- **Sphericity,  $\Psi$**

$$\Psi = \frac{\text{surface area of sphere having same volume as particle}}{\text{surface area of particle}}$$





## Particle Shape vs. Characteristic Dimension, $d_{ES}$

- Particle behavior in physical separations will depend on surface area and volume as well as size and other factors.
  - Surface area and volume differ broadly in their dependence on “particle size” according to its shape.
  - Additional factors include the phase density difference, liquid viscosity, surface tension and electrokinetic phenomena (zeta potential).
- Using the equivalent sphere diameter  $d_{ES}$  as the characteristic dimension:
  - $d_{ES}$  = diameter of sphere having projected area equal to particle
  - Surface area =  $f * (d_{ES})^2$
  - Volume =  $k * (d_{ES})^3$

Particle Type or Shape	f	k	$\Psi$
Sphere	$\pi$	$\pi / 6$	1.0
Rounded particles, water-worn sands, atomized metals, fused flue dust	2.7 – 3.4	0.32 – 0.41	0.82
Angular particles of crushed minerals	2.5 – 3.2	0.2 – 0.28	0.66
Flaky particles: talc, gypsum	2.0 – 2.8	0.12 – 0.16	0.54
Thin flaky particles: mica, graphite, aluminum	1.6 – 1.7	0.01 – 0.03	0.22

Ref: Rushton, Ward, Holdich

# Material Parameters: Particle Size Distributions

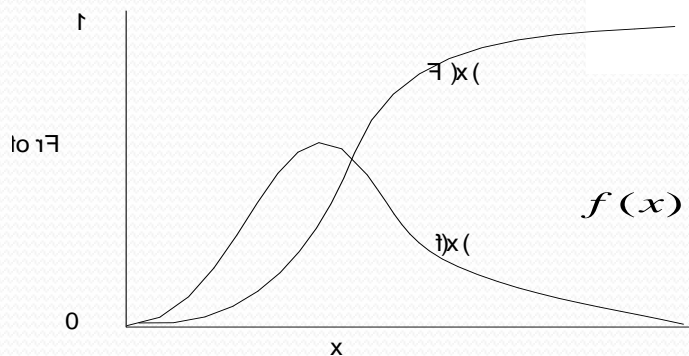
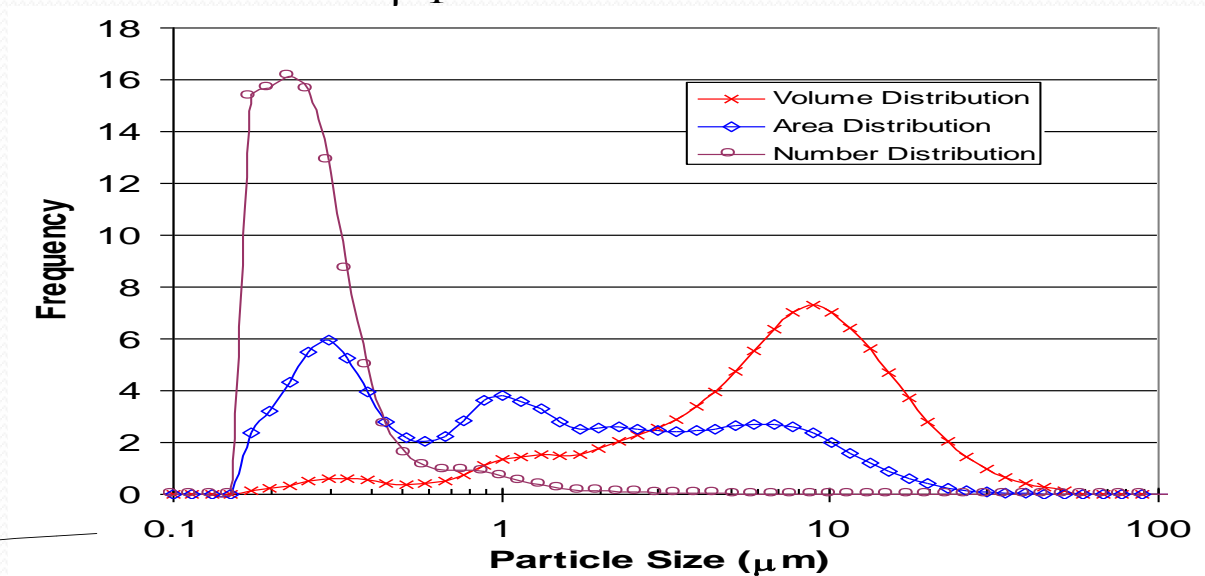
- Number Distribution
- Area Distribution
- Volume Distribution

$$f_{i,n} = \frac{n_i}{\sum_{j=1}^m n_j}$$

$$f_{i,n} = \frac{n_i d_i^2}{\sum_{j=1}^m n_j d_j^2}$$

$$f_{i,n} = \frac{n_i d_i^3}{\sum_{j=1}^m n_j d_j^3}$$

Same particle size distribution data expressed for different bases.

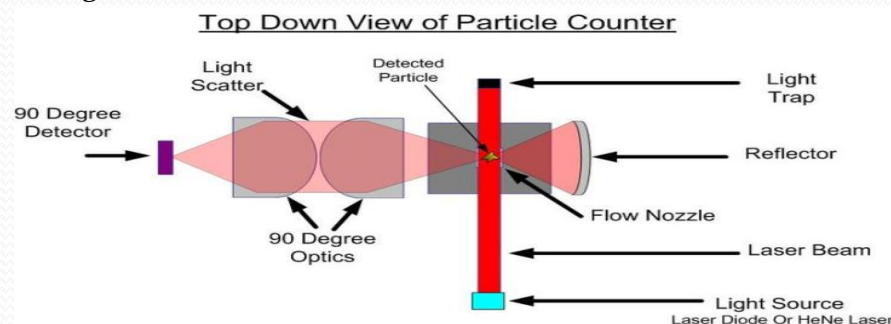


$$f(x) = \frac{dF(x)}{dx}$$

Frequency (f) vs.  
Cumulative (F)  
Distribution

# Material Parameters: Optical Particle Size Measurement

- Single-Particle Optical Sensing (SPOS = Particle Counting)
  - Passage of single particle through a sensing zone causes a detected pulse, the magnitude of which depends on the mean diameter of the particle and the physical principle of detection
    - Light scattering or light obscuration (blockage)
    - PSD constructed one particle at a time by referring to a standard calibration curve derived from uniform particles of known diameters.
    - Coincidence of particles in the sensing zone must be avoided (dilute).
    - Superior resolution and accuracy, wide dynamic range.



- Ensemble Techniques
  - Large no. of particles of different size contribute simultaneously to the measured signal.
  - Most common methods of detection:
    - Sedimentation/turbidity: optical or x-ray transmission through sample at a fixed location in the suspension. Most useful range:  $1 - 10 \mu$
    - Fraunhofer laser diffraction: angular distribution of diffracted light intensity where the characteristic angle of diffraction decreases with increasing particle size. Most useful range:  $0.1 - 3000 \mu$
    - Inherently limited accuracy and resolution but versatile. Raw detected signal must be inverted using an analysis algorithm to estimate PSD.
      - Assume a PSD using few parameters then minimize the error between measured data and the calculated fit by adjusting parameters.
      - Limitations imposed by signal/noise ration of raw data coupled with “ill-conditioned” nature of the deconvolution algorithm.

# Altering Effective Particle Size to Promote Separation

- Coagulation

- Agglomeration of primary particles into assemblies up to 1 mm
- Relative adhesion of particles due to molecular or atomic forces
  - attractive forces: van der Waals', hydrogen bonding
  - repulsive force: formation of electrical double layer
  - net repulsive force between particles increases with concentration
- Frees liquid by destabilizing suspension
  - adding non-adsorbing electrolyte to change distribution of solution ions
  - altering chemical charge on the particle surface with adsorbed ions or charged polymers
- Promoted by inorganic or organic chemical additives
  - common inorganic ions:  $\text{Al}^{+3}$ ,  $\text{Fe}^{+3}$ ,  $\text{Ca}^{+2}$
  - natural clays having negative charge can coagulate a positive colloid

- Flocculation

- Agglomeration and interconnection of primary particles into loose assemblies up to 1 cm in size
- Long-chain polymeric molecules form bridges between particles
  - natural substances (e.g. gelatin, starch, phenolic tannins, guar gum) and synthetic polyelectrolytes (anionic, cationic, non-ionic)
  - polymer flocculants are characterized by molecular wt. and charge
  - flocculating action is irreversible so high shear rates must be avoided after initial formation of flocs
- In proper dosages, flocculants adhere to ~50% of solid surfaces
  - initial agitation needed for uniform dispersal
  - flocculants remain with solid phase for disposal
  - excessive dose can coat entire particle surface, stabilizing suspension

- Rate of Aggregation Depends on Two Mechanisms

- Perikinetic (Brownian motion) vs. Orthokinetic (bulk fluid motion)
- Relative measure of forces: 
$$\frac{\text{orthokinetic rate}}{\text{perikinetic rate}} = \frac{G\mu d_p^3}{2kT}$$

where

k = Boltzmann const.

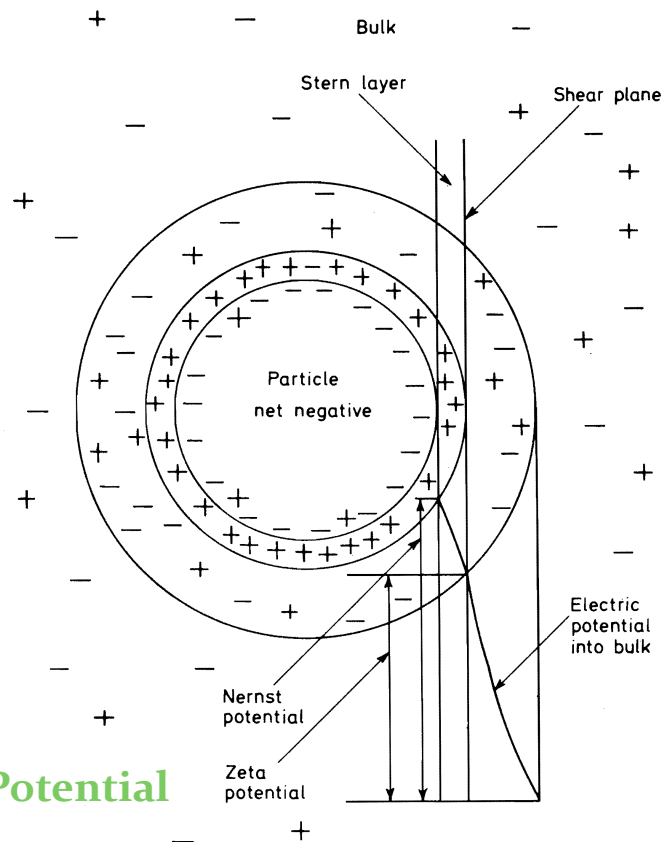
G = shear rate

T = temperature

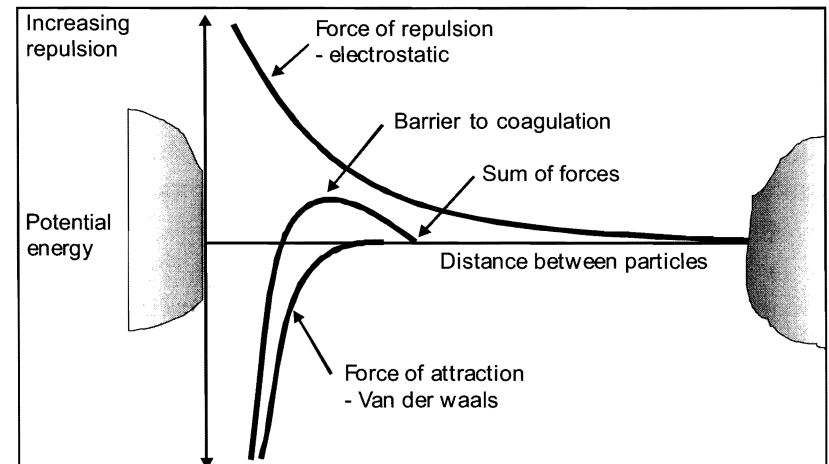
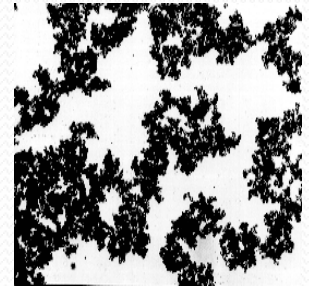
# Material Parameters: Colloidal Model for Particle Charge Effects

- Surface charges on particles can affect the rate and extent of different physical separations.
- Magnitude of actual charge is not easily measured but the **effective charge in suspension can be characterized by the particle's zeta potential ( $\zeta$ )**.
- For aqueous suspending liquid, **zeta potential varies with pH and ionic strength**. It is less meaningful for non-polar organic liquids.
- Instrument analyzers include PSS NICOMP™ 380ZLS, Coulter DelsaMax™

## Double Layer Formation with Zeta Potential



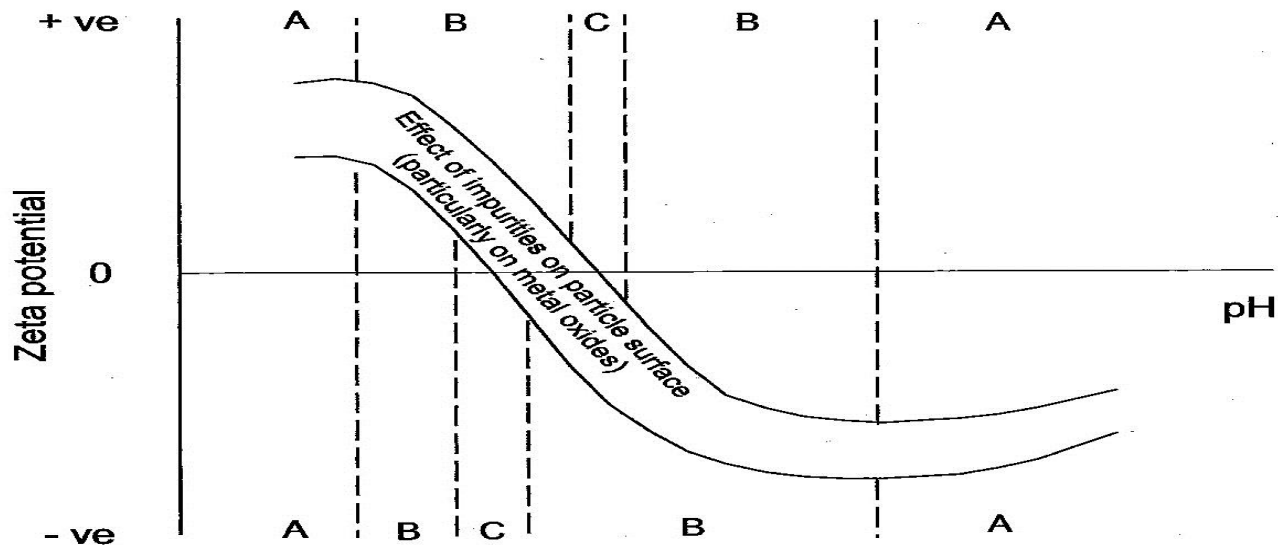
Lowering Zeta potential by adding non-adsorbed electrolyte ions to bulk solution can compress double layer and promote agglomeration of solids with release of solids-free liquid.



Zeta Potential



## Variable Impact of Solution pH/Ionic Strength on Particle Behavior



Ref: Wakeman, Tarleton, Principles of Industrial Filtration, Elsevier, 2005.

- A: High ionic strength causes compression of double layer. Intermediate settling and possibly low filtration rates. Intermediate residual liquid in cake or sediment.
- B: Higher zeta potential, greater repulsion between particles. Well dispersed stable suspension. Slower separation rates. Lower cake or sediment moisture.
- C: Close to the isoelectric point (zero zeta potential), so little repulsion between particles. Particles tend to aggregate. Faster settling rates, more rapid rates of filtration and cake formation, slightly higher moisture in cake or sediment.
- Wash liquor or solvent can change distribution of ionic species and reduce porosity of cake or sediment making wash more effective in purging residual liquor.

# Material Parameters: Altering Solid Phase via Filter Aids

- Different Purposes of Filter Aid by Method of Addition
  - Body Feed: intended primarily to improve permeability and specific filtration rates.
  - Precoat: intended above all to improve filtrate clarity and release of cake from the filter cloth.
- Properties of 3 Major Types of Filter Aids Classified by Origin

Summary of major types of filter aids (Purchas, 1981). (\*Water permeability ratio relative to Dicalite 215, which has a bed permeability of about  $2 \times 10^{10} \text{ m}^2$ )

	Dicalite diatomite	Dicalite perlite	Solka-Floc
Composition	silica	glassy silica	cellulose
Number of grades	15	8	8
Range of relative flow rates*	1–23	1.7–9.3	5–23
Specific gravity	2.25–2.33	2.34	1.5
Wet cake density	0.32–0.38 g cm <sup>-3</sup>	0.24–0.34	0.17–0.34
Retention of coarse screens	good	good	excellent
Solubility (at room temperature)			
– in alkalis	slight in dilute	slight in dilute	none in dilute or strong
– in acids	slight in dilute	slight in dilute	none in dilute
Prime advantages and applications	General use for maximum clarity. Reduced dosage on pressure and vacuum filters.	Good on rotary filters.	Excellent for pre-coating coarse screens. Highest purity for absorption of oil from condensate and removal of iron from caustic.

# Material Parameters: Liquid Properties

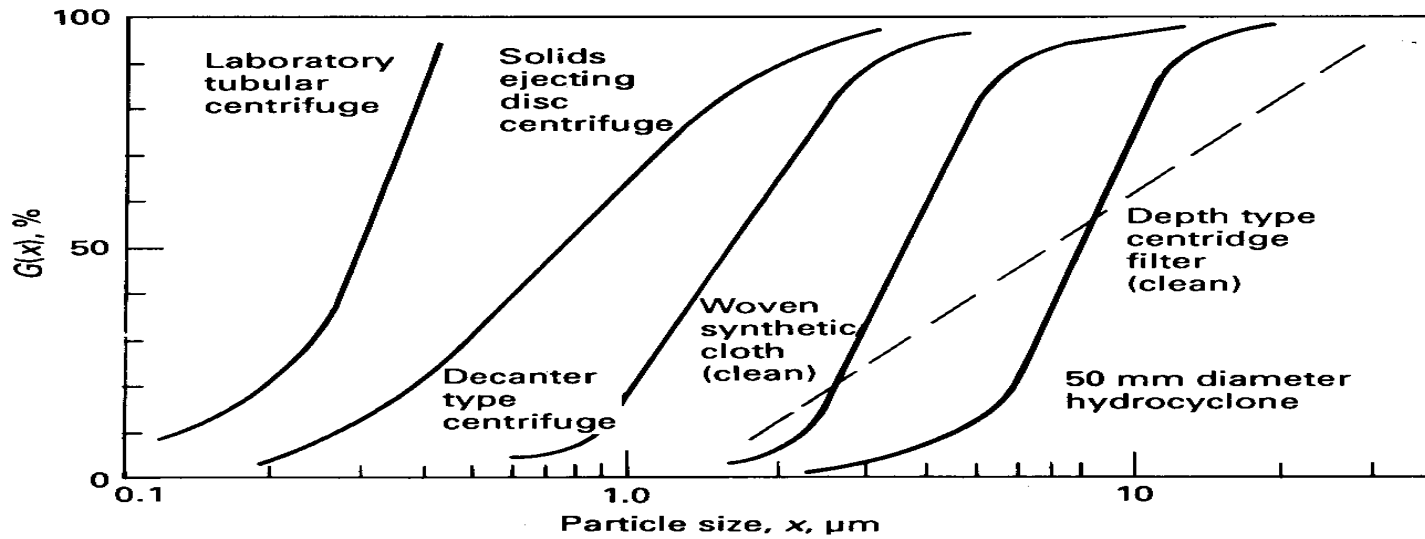
- Liquid Viscosity (dynamic, kinematic)
  - Key transport property in the liquid momentum balance.
  - Temperature dependence can present a significant factor in specific filtrate flow rate.
  - Easily measured using capillary, Couette/Searle or rotational (Brookfield) viscometers.
  - Categories of filtration behavior:
    - 0.2 – 0.8 cP: ultra-low viscosity facilitates all physical phase separations.
    - 0.8 – 10.0 cP: low viscosity allows judicious use of most technologies.
    - 10 – 100 cP: moderate viscosity favors pressure and depth filtration over vacuum filtration and sedimentation.
    - 100 – 1000 cP: high viscosity practically limits technologies to pressure filtration (cake, depth, thin layer, straining) and mechanical expression.
    - > 1000 cP: ultra-high viscosity requiring specialized high-pressure , straining devices (e.g. screen changers, multi-layer, high-area metal screens).
- Liquid Vapor Pressure
  - Non-aqueous solvents typically require containment regardless of magnitude for EH&S reasons.
  - > 0.5 bar for any liquid can defeat use of vacuum filtration because of high vapor traffic incurred.
- Solution pH and Ionic Strength
  - Affects the extent of flocculation or dispersion of solid particles.
  - Dictates corrosion potential, acceptable materials of construction (MOC) for vessels, media, seals, etc.

# Process and Multiphase Parameters

- Operating Temperature
  - Liquid dynamic viscosity (if Newtonian) and vapor pressure are surprisingly strong functions of temperature.
  - Restricts the selection of filter media.
    - Most available materials are polymers having relatively low glass transition temperatures.
    - Ceramic and metal filter media are relatively insensitive to temperature and pressure.
  - Affects the choice of structural materials for SLS equipment (e.g. vessels, supports, etc.).
- Interfacial Tension
  - Function of the surface energy where solid particles are wet by the suspending liquid.
  - Affects the efficiency of displacement washing of porous solid deposits (e.g. filter cake).
  - Capillary Suction Timer (CST) can be used to evaluate relative drainage rates of liquid from solids after coagulant addition.
- Permeability
  - For Filtration: Relates the drag force created by a difference in local phase velocities to the local pressure gradient.
  - Filter Medium is a key factor in determining the flow permeability of a porous deposit.
- Effective Suspension Viscosity
  - Limits solids concentration in crossflow filter design.
- Grade Efficiency Curve: Solids Removal Efficiency as Function of Particle Size

## Process and Multiphase Parameters: Grade Efficiency Curve, $G(x)$

- Describes solids removal efficiency as a function of particle size:



- For sedimentation devices,  $G(x)$  is constant for a set of operating conditions and is relatively independent of the feed solids size distribution (e.g. numerous fines will pass through)
- For filtration devices,  $G(x)$  is strongly influenced by the quantity of solids accumulated on or in the porous filter medium.
- Position and shape of  $G(x)$  is useful for comparing clean filter media. Sharp cut-off used for size classification.
- “Cut Size” specification reduces  $G(x)$  curve to a single number for comparison
  - $x_{50}$  denotes the size ( $x$ ) for which 50% of solids by weight pass to the overflow, centrate or filtrate.



# Multiphase Material Parameters: Filter Medium Type and Properties

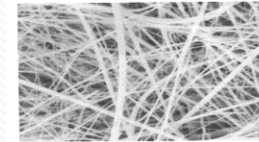
- Multifilament Yarn, Monofilament Yarn, Random Fiber Felt



Multifilament



Monofilament



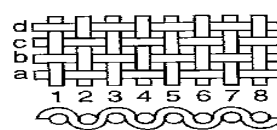
Random Fiber

- Effect of Yarn Type on Cloth Performance (Purchas)

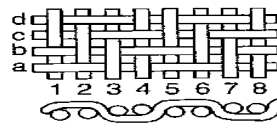
decreasing ↓

Max. filtrate clarity	Min. flow resistance	Min. cake moisture	Best cake discharge	Max. cloth life	Least tendency to blind
Staple	Monofil	Monofil	Monofil	Staple	Monofil
Multifil	Multifil	Multifil	Multifil	Multifil	Multifil
Monofil	Staple	Staple	Staple	Monofil	Staple

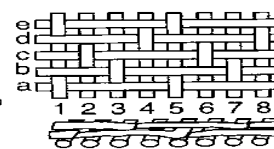
- Basic Types of Weaves



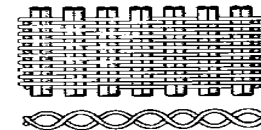
Plain



Twill



Satin



PRD

- Impact of Cloth Weave on Performance (Svarovsky, ed.) for 40-μ nominal retention with each weave.

decreasing ↓

Max. filtrate clarity (without cake) (?)	Min. flow resistance (?)	Min. cake moisture (?)	Easiest cake discharge (reversed for sticky cake)	Max. cloth life (equipment dependent)	Least tendency to blind (?)
Satin	Plain	Plain	Satin	Satin	Plain
Twill	PRD	PRD	Twill	Twill	PRD
PRD	Twill	Twill	Plain	PRD	Twill
Plain	Satin	Satin	PRD	Plain	Satin

# Principal Mechanisms for Accomplishing Solid-Liquid Separations

- All Legitimate Models Reduce to the Mass and Momentum Balances for Each Phase.
- Size Exclusion of Solid Phase with Hydraulic Pressure-Driven Flow
  - Straining
  - Thin Layer Crossflow (Microfiltration, most Ultrafiltration)
  - Cake Filtration (Pressure and Vacuum)
  - Depth Filtration (Cartridge, Deep Bed)
- Body Force Acting on Different Phases To Produce Relative Motion
  - Sedimentation (based on mass concentration)
    - Gravity Settling and Flotation
    - Centrifugal Decanting
- Electro-Chemical Adsorption under Hydraulic Pressure-Driven Flow
  - Depth Filtration (Deep Bed, Cartridge)
  - Magnetic Separation
- Less Common, Evolving Technologies
  - Mass Diffusion Superimposed on Pressure-Driven Convective Flow
    - Some Ultrafiltration, Nanofiltration
  - Osmotic Pressure (Solids Deliquoring)
  - Ultrasound (Solids Deliquoring)

# Physical Principles: Single Particle Sedimentation

- Stokes' (laminar) settling,  $Re_p \leq 0.2$ 
  - Infinite liquid with no particle interaction, practically implying solids volume%  $\leq 0.5$
- Particle Reynolds' No. ( $Re_p$ )
  - Particle of size  $x$  at velocity  $u$  relative to liquid of density  $\rho_L$  and viscosity  $\mu$
- Terminal settling velocity  $u_T$ 
  - For particle having Stokes' diameter  $d_{st}$  and acceleration ( $RCF \bullet g$ ) under applied body force equals:
  - For gravity,  $RCF = 1$ .
  - Model has lower limit on particle size of 1 micron due to Brownian diffusion.
  - Upper limiting size  $x_{CR}$  for  $Re_p \leq 0.2$  can be calculated.
- Practical effective phase separation by gravity alone requires 50 kg/m<sup>3</sup> density difference.

$$Re_p = \frac{\rho_L u x}{\mu}$$

$$u_T = \frac{(\rho_s - \rho_L)(RCF \bullet g)d_{st}^2}{18\mu}$$

$$x_{CR} = \sqrt[3]{\frac{3.6\mu^2}{g(\rho_s - \rho_L)\rho_L}}$$

# Physical Principles: Multi-Particle Sedimentation

- Hindered Settling at Higher Concentrations
  - When return flow of liquid displaced by settling particles is uniformly distributed (i.e. all but monosized suspensions), settling velocity declines with increasing concentration.
  - Richardson and Zaki showed that hindered terminal velocity  $u_H$  is linked to particle  $Re_T$  for that velocity by a function of the liquid volume fraction ( $\epsilon$ ) of suspension,  $f(\epsilon) = \epsilon^{2.65}$ .
    - this dependence on void fraction can be represented more generally as:

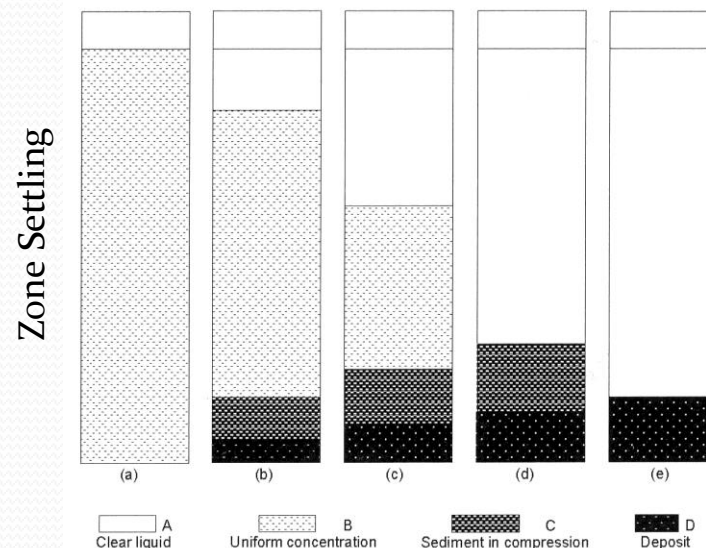
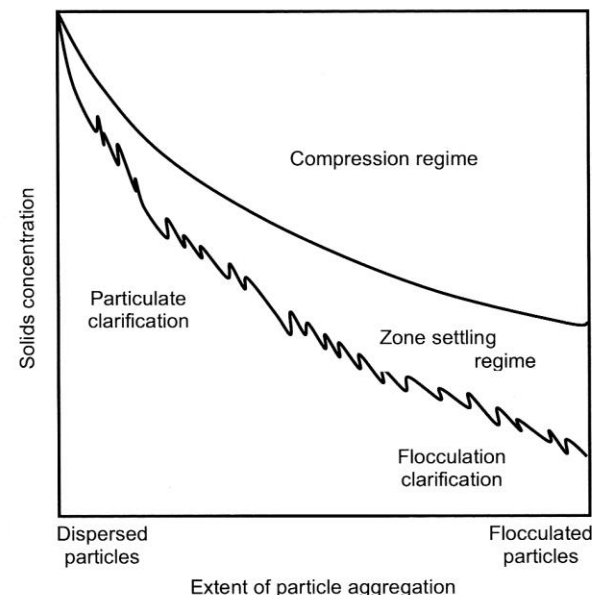
$$u_H = u_T \epsilon^2 f(\epsilon) = u_T \epsilon^n$$

$Re_T < 0.2$	$n = 4.6 + 20 x/D$
$0.2 < Re_T < 1$	$n = (4.4 + 18 x/D) Re_T^{-0.03}$
$1 < Re_T < 200$	$n = (4.4 + 18 x/D) Re_T^{-0.1}$
$200 < Re_T < 500$	$n = 4.4 Re_T^{-0.1}$
$Re_T > 500$	$n = 2.4$

where  $Re_T$  is based on particle diameter and  $D$  equals sedimentation vessel diameter. These correlations apply to particulate settling and do not hold if flocculation occurs.

# Physical Principles: Sedimentation Regimes

- 4 Regimes of Sedimentation
  - Particulate and Flocculated Clarification
  - Zone Settling
  - Sediment Compression
- Analysis differs with dependence on residence time.
  - Performance of particulate clarification depends only on overflow rate.
  - Performance of flocculating suspensions at high solids concentrations depends on time.
  - Residence time provided by settling distance (height).
- Zone Settling Behavior
  - If their size range does not exceed 6:1, particles below an interface with clear liquid can settle at a uniform velocity regardless of size at a rate that depends on concentration, a phenomena called “Zone Settling” .
  - Concentration at which zone settling occurs depends on the material, its interparticle cohesion and the state of flocculation.
  - The terminal velocity of flocs  $u_H$  and value of parameter  $n$  can be determined by a linear-log plot of experimental data.,  $u_H$  vs.  $\epsilon$ . Floc diameter can be estimated from the intercept at  $\epsilon = 1$ , which equals Stokes’ terminal velocity.





# Physical Principles: Particulate Gravity Sedimentation Design

- Two important factors to reconcile in sizing gravity settler:
  - Area required to settle solids and clarify overflow
  - Area required to concentrate solids in the underflow
- Clarifier Design for Particulate Settling
  - Liquid rise rate to overflow must be less than hindered settling velocity:  $(Q_O/A) < u_H$ , or  $A > (Q_O / u_H)$
  - Rise rate correlated to overflow TSS by decanting short settling tube for various time intervals.
  - Conventional design factor of 2 applied to rise rate, i.e.  $u_{\text{design}} = 0.5 u_H \geq 0.5 (Q_O/A)$ .
- Thickener Design
  - At steady state, the rate of solids passing through the thickener (solids flux,  $G_T$ ) at concentration  $C$  equals the rate at which solids are discharged in the underflow at concentration  $C_u$ :

$$G_T = \frac{Q_u}{A} C_u$$

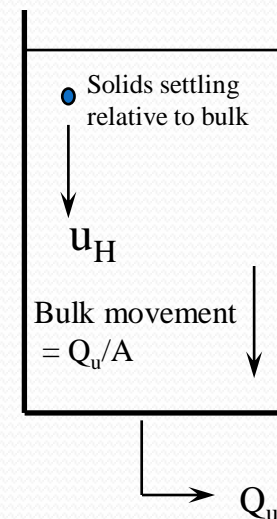
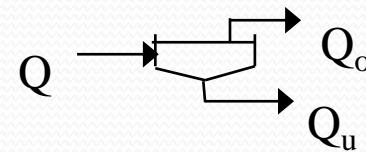
- Total solid flux also equals the sum of relative settling flux and bulk flux:

$$G_T = u_H C + \frac{Q_u}{A} C = \left( u_H + \frac{Q_u}{A} \right) C$$

- Combining the expressions for solids flux  $G_T$  yields the design equation:

$$G_T = \frac{u_H}{\frac{1}{C} - \frac{1}{C_u}}$$

- Procedure:
  - Perform batch settling tests at several solids concentrations between the feed and underflow concentrations to determine hindered settling rate  $u_H$  at each concentration.
  - Calculate total flux  $G_T$  for each combination of  $u_H$  and  $C$ .
  - Maximum solids throughput in a thickener is governed by the concentration layer having the lowest solids flux.
  - Minimum solids flux  $G_{\min}$  is then used to set the minimum cross-sectional area ( $A_{\min}$ ) of the thickener.



# Physical Principles: Centrifugal Sedimentation, Derivation of $\Sigma$

- Centrifugal terminal velocity of particle having size  $x$ , radial position  $R$  and angular speed  $\omega$  can be expressed as
- Integrating terminal velocity expression from initial radius  $R_0$  to  $R$  at time  $t$  yields a means of calculating the settling time for a particle of size  $x$  to move a radial distance  $(R - R_0)$ :
- For volumetric flow rate  $Q$  to a centrifuge bowl of length  $L$  and radius  $r_B$  with pool surface at radius  $r_p$ , bowl residence time is determined. For that time, the limiting trajectory to capture a particle of size  $x$  implies that particles starting at radius  $r$ , where  $r_p < r < r_B$ , will be captured:
- For 50% overall efficiency,  $G_d = 0.5$ , and simplifying  $\Sigma$ , it becomes Ambler's Sigma factor for tubular bowl centrifuges which is a function of machine dimensions and rotating speed:
- Scale-up to a larger but similar sedimenting centrifuge is based on pilot results, physical properties of the suspension and the characteristic  $\Sigma$ -factor for that decanting centrifuge geometry where  $Q$  is the feed rate of process suspension,  $U_g$  is obtained from Stokes' equation,  $g$  is gravitational acceleration, and  $\Delta\rho$  equal to density difference (solid – liquid).

$$U_{TC} = \frac{x^2 \Delta\rho R \omega^2}{18\mu}$$

$$\frac{R(t)}{R_0} = \exp\left[\frac{x^2(\rho_s - \rho_L)\omega^2 t}{18\mu}\right] = \exp[x^2 B t]$$

$$\frac{r}{r_B} = \exp\left[\frac{-\pi B L (r_B^2 - r_p^2) x^2}{Q}\right]$$

$$\Sigma_{\text{tubular\_centrifuge}} = \pi \omega^2 L \left[ \frac{3r_B^2 + r_p^2}{2g} \right]$$

$$2U_g \Sigma = Q$$

$$U_g = \frac{x_{50}^2 \Delta\rho g}{18\mu}$$

# Physical Principles: Centrifugal Sedimentation Scale-Up Using $\Sigma$

- Sigma ( $\Sigma$ ) Factor Scale-Up Method

- Experimental pilot tests establish the performance of a specific centrifuge type and size.

- Centrifuge Scale-Up becomes: 
$$\left[ \frac{Q}{\Sigma} \right]_{pilot} = \left[ \frac{Q}{\Sigma} \right]_{full-scale}$$

- Features of  $\Sigma$ :

- Centrifuge dimensions, rotational speed and geometric configuration are captured in  $\Sigma$ .
- Expressions derived for  $\Sigma$  imply a grade efficiency  $G(x)$  equal to 0.5. Although derived for  $G(x) = 0.5$ , similitude applies to any efficiency common to both scales.
- Particle and liquid properties summarized in the terminal settling velocity  $U_{gz}$  under gravity for median particle size (i.e. cut size  $x_{50}$ ).
- $\Sigma$  has dimension of area and in theory represents the area of equivalent gravity settler (but comparison ignores factors that are significant for gravity settling).
- Calculated values of  $\Sigma$  ignore end effects and particle re-entrainment.

- Dimensionless form of  $\Sigma$  design equation:  $Stk_{50} Fr = 0.5$

- $D$  is a characteristic dimension (e.g. bowl diameter) that cancels out

$$Stk_{50} = \frac{x_{50}^2 \Delta \rho}{18 \mu} \frac{Q}{\Sigma D} \quad Fr = \frac{D g \Sigma^2}{Q^2}$$

- Key elements in a sedimentation (decanter) centrifuge specification:

- Solids recovery or clarity of the overflow (most frequent)
- Solids underflow concentration
- Solids loading
- Scroll torque and/or drive motor power; mechanical limits of machinery (materials, reliability).
- Shear effects in sedimenting centrifuge are sufficiently weak to experiment with flocculating agents.

# Physical Principles: Common Decanter Centrifuges $\Sigma$

- Application of  $\Sigma$  Analysis to Common Types of Decanter Centrifuges

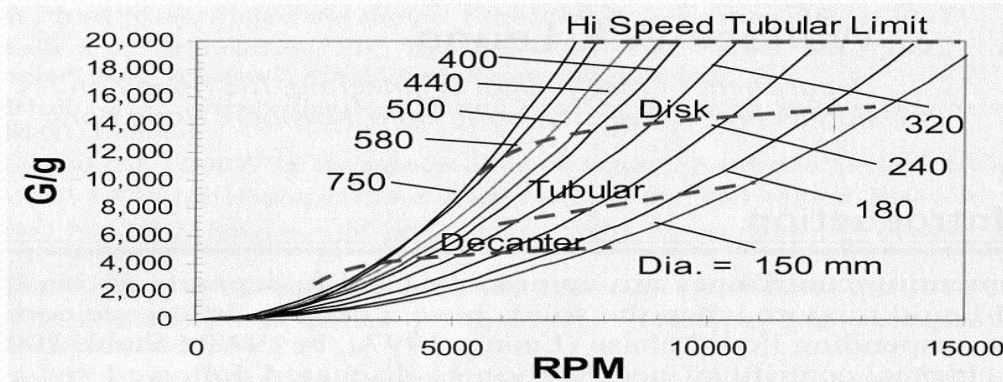
- Allows comparison of flow capacities at specified removal efficiencies among centrifuges having similar geometric and hydrodynamic features.
  - General form:  $\Sigma = (\pi\omega^2 / g) K$  (Note that phase properties are not factors in  $\Sigma$ )
  - Sigma is a function of centrifuge dimensions and speed and is best used as an index of centrifuge size. Flow capacity becomes a function of  $\Sigma$  and cut size ( $x_{50}$  or other).
  - Sigma concept ignores effects of Brownian diffusion and convection currents and flocculation.
- For conical solid bowl scroll decanter centrifuge rotating at speed  $\omega$  with bowl radius  $r_B$ , pond radius  $r_P$ , conical section length  $L_{cone}$ , and cylindrical section length  $L_{cyl}$ :

$$\Sigma_{conical\_solid\_bowl} = \frac{\pi\omega^2}{g} \left[ L_{cyl} (1.5r_B^2 + 0.5r_P^2) + L_{cone} \left( \frac{r_B^2 + 3r_B r_P + 4r_P^2}{4} \right) \right]$$

- For a disk stack centrifuge rotating at speed  $\omega$  with (n+1) disks (n channels) of inner radius  $r_i$  and outer radius  $r_o$  inclined at an angle  $\theta$  with the vertical axis:

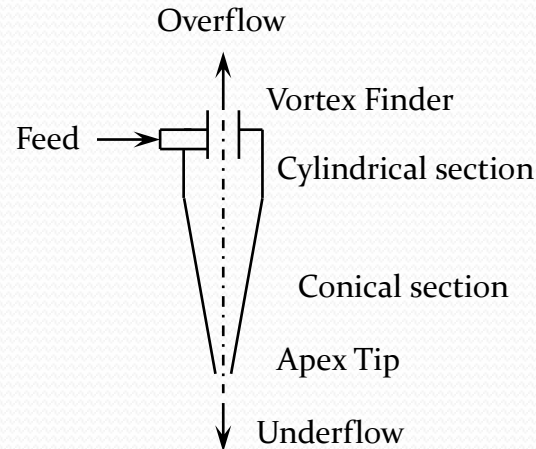
$$\Sigma_{disk\_stack} = \frac{\pi\omega^2}{g} \frac{2n}{3} (r_o^3 - r_i^3) \cot \theta$$

- Comparative ranges of force at speed for different sedimentation centrifuges



# Physical Principles: Hydrocyclone Sedimentation

- Operating Principle
  - Pumping liquid into tangential port of stationary device generates centrifugal force which separates phases.
- Hydrocyclone Characteristics
  - Diameters: 10 mm – 2.5 m (0.5 – 100 in.)
  - Cut Sizes: 2 – 250  $\mu$
  - Capacity: 0.1 – 7200 m<sup>3</sup>/h (0.5 – 31000 gpm)
  - Pressure Drop: 0.34 – 6 bar (5 – 90 psi)
  - Underflow Concentration: up to 30% by volume





# Physical Principles: Hydrocyclone Dimensionless Model

- Modeling Hydrocyclone for Low Feed Solids Concentration (< 1% Vol.)
  - Assumes negligible particle interaction or alteration of flow pattern, low underflow to throughput ratio with no effect on cut size  $x_{50}$
  - 3 dimensionless groups ( $Stk_{50}$ ,  $Re$ ,  $Eu$ ) relate 6 process variables:  $x_{50}$ , hydrocyclone diameter  $D$ ,  $\mu$ ,  $\rho_L$ ,  $\rho_S$  and feed suspension flow rate  $Q$ 
    - $Re = (v D \rho) / \mu$ , where  $v$  is characteristic velocity based on cross-sectional area of cylindrical section
    - $Eu = \Delta P / (\rho v^2 / 2) = f(Re)$  is a pressure loss factor where  $\Delta P$  is determined experimentally
    - $Stk_{50} = f(Re, \Delta \rho / \rho)$
- Studies show that  $\Delta \rho / \rho$  has no effect, so  $Stk_{50}$  and  $Eu$  depend only on  $Re$  and geometrically similar hydrocyclones can be scaled up.
- $Stk_{50} Eu = \text{constant}$  determined from dimensions, properties and experimental performance.
  - Rietema residence time model:  $Stk_{50} Eu = 0.0611$ ,  $Eu = 24.38 Re^{0.3748}$
  - Bradley equilibrium orbit model:  $Stk_{50} Eu = 0.1111$ ,  $Eu = 446.5 Re^{0.323}$
- Design Procedure
  - Determine experimental parameters with small ( $\leq 50$  mm) hydrocyclone then scale up separation for geometrically similar, larger units in production.
  - Assess process performance from models and geometries available. Optimize hydrocyclone diameter, number and pressure drop.

$$v = \frac{4Q}{\pi D^2}$$

$$Stk_{50} = \frac{x_{50}^2 (\rho_S - \rho_L) v}{18 \mu D}$$



## Physical Principles: Flow Through a Packed Bed

- Darcy's Law (empirically derived):

- $Q$  = volumetric flow rate ( $\text{m}^3/\text{s}$ )
- $A$  = face area of bed, normal to flow direction ( $\text{m}^2$ )
- $L$  = bed depth (m)
- $\Delta P$  = pressure drop across the bed ( $\text{N}/\text{m}^2 = \text{Pa}$ )
- $\mu$  = liquid viscosity ( $\text{N}\cdot\text{s}/\text{m}^2 = \text{Pa}\cdot\text{s} = \text{poise} = 100 \text{ cP}$ )

$$\frac{Q}{A} = \frac{\Delta P}{\mu L} K$$

- Kozeny - Carman Equation:

- Definition of terms:
  - $K_C$  = Kozeny constant
  - $\varepsilon$  = bed void volume fraction or porosity (dimensionless)
  - $S_o$  = volume specific bed surface = (total particle surface / volume of particles)
  - $Q/A$  = superficial velocity = (porosity  $\varepsilon$ ) \* (interstitial velocity)
- Models porous bed as a bundle of capillary tubes oriented at  $45^\circ$  to inlet face area
- Derived and valid for laminar flow ( $Re_p \leq 1$ )
- Constant  $K_C$  depends on particle size, shape and porosity;  $K_C \sim 5$  at low porosity
- Equation's assumptions break down at higher porosities ( $\varepsilon > 0.7$ )
- Accurately describes flow of dilute suspension through a deep bed filter prior to solids accumulation.

$$\frac{Q}{A} = \frac{\Delta P}{\mu L} \frac{\varepsilon^3}{K_C (1 - \varepsilon)^2 S_o^2} = \frac{\Delta P}{\mu L} \frac{D_p^2 \varepsilon^3}{36 K_C (1 - \varepsilon)^2}$$

# Physical Principles: Cake Filtration – Conventional Analysis

- Conventional Cake Filtration Model

- Two Serial Resistances to Flow Formed by Porous Media: Cake + Cloth/Screen
- General filtration eq. derived from Darcy's Law for constant pressure drop  $\Delta P$ :
- Definition of terms:

$$\frac{Q(t)}{A} = \frac{dV}{Adt} = \frac{\Delta P}{\mu \left[ \alpha c \left( \frac{V(t)}{A} \right) + R_M \right]}$$

$$c = \frac{\rho_L s}{1 - ms}$$

- $\alpha$  = specific cake resistance based on mass of dry cake solids (m/kg)
- $c$  = mass of dry cake solids per volume of filtrate (kg/m<sup>3</sup>)
- $m$  = (mass of wet cake) / (mass of dry cake)
- $s$  = solids mass concentration in the feed suspension
- $R_M$  = filter medium resistance (m<sup>-1</sup>)
- $V(t)$  = cumulative volume of filtrate collected at time  $t$  (m<sup>3</sup>)
- $A$  = filter area (m<sup>2</sup>)

- Assumptions:

- All feed suspension forms wet filter cake or filtrate, i.e. no residual slurry
- Solids concentration in filtrate is insignificant
- Flow resistances act in series with no interaction
- Cake resistance is characteristic of a unit mass of cake

- To calculate volume as function of time for constant pressure filtration, integrate assuming cake resistance depends only on pressure and medium resistance remains constant, then rearrange to get parabolic filtration equation for so-called incompressible cakes.

$$\frac{t}{V} = \frac{\mu c \alpha}{2 A^2 \Delta P} V + \frac{\mu R_M}{A \Delta P}$$

- For constant rate filtration,  $(dV / dt) = V/t = \text{constant}$ , yielding

$$\Delta P = \left( \frac{\mu c \alpha}{A^2} \right) \left( \frac{V}{t} \right) V + \mu R_M \left( \frac{V}{t} \right)$$

- For “compressible” cakes,  $\alpha$  is postulated as function of differential pressure and integrated over cake pressure drop to obtain average  $\alpha$ .
- To handle negative intercepts, Darcy's Law is used for the initial portion of filtration.

# Physical Principles: Cake Filtration – Multiphase Analysis

- Volume-Averaged Conservation of Momentum for Cake Filtration

- Dimensional analysis reduces to force balance relating pressure, viscous interfacial drag and body forces.
  - Viscous drag can be represented in Darcian form as a product of porosity, the velocity difference between phases, and reciprocal permeability.
  - Centrifugal force and gravity would appear as body forces in momentum balances.
- Separate, averaged equations of motion are obtained for liquid and solid phases.
- Applying boundary condition at interface between cake and medium, the averaged equation of liquid motion in one direction (z) yields design equation:

$$\left( \frac{\mu L_C}{A_M \Delta P_C} \right) \frac{dV}{dt} = K_O \left( \frac{\partial P^*}{\partial \xi} \right)_{\xi=0}$$

- Definition of Terms

- $L_C$  = cake length at time t
- $\Delta P_C$  = hydraulic pressure drop across the cake alone
- $A_M$  = area normal to flow direction at  $z = 0$ , i.e. surface of filter medium
- $\xi$  = dimensionless distance =  $z/L(t)$ ,  $z = 0$  at the filter medium
- $\partial P^*/\partial \xi$  = dimensionless pressure gradient,  $P^* = P/\Delta P_C$
- $K_O$  = permeability at the cake-medium interface

- Reciprocal Rate Equation

- For material ratio  $G(s, \langle \varepsilon \rangle) = (L_C A' / V)$  derived from an overall material balance where  $\langle \varepsilon \rangle$  is average cake porosity,  $s$  is feed solids concentration, and both solid and liquid are intrinsically incompressible, rearranging yields:

$$\frac{dt}{dV} = \left( \frac{\mu G(s, \langle \varepsilon \rangle)}{A_M A' \Delta P_C} \right) \left( \frac{1}{K_O \left[ \frac{\partial P}{\partial \xi} \right]_0} \right) V \quad \text{Material Ratio} \quad G = \frac{\rho_L s}{\rho_s (1 - \langle \varepsilon \rangle)(1 - s) - \rho_L \langle \varepsilon \rangle s}$$

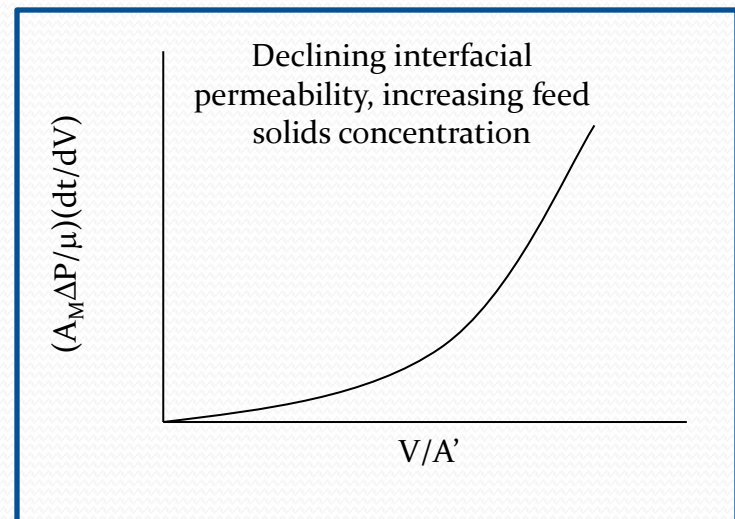
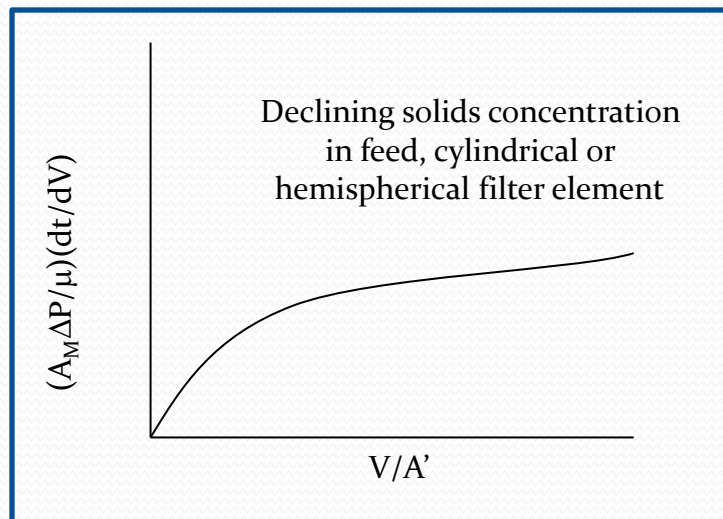
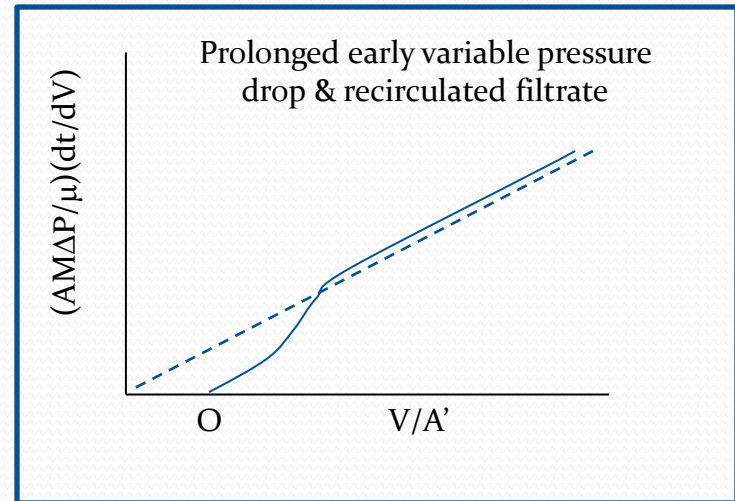
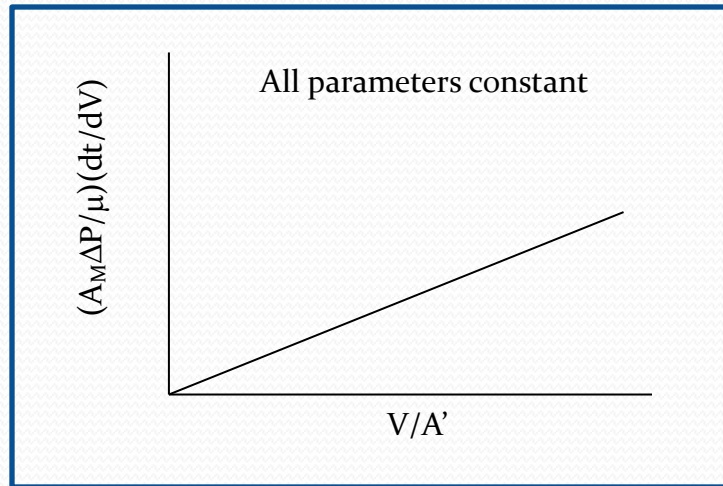
- Implications

- Interface between the filter cake and medium governs the filtrate flow rate at any instant for given values of cake pressure drop, filter area and liquid viscosity.
- Interfacial product must be characterized as a function of filtrate volume or cake length.

# Interpreting Reciprocal Filtrate Flow Rate for Cake Filtration in One Direction

$$\frac{dt}{dV} = \left( \frac{\mu G(s, <\varepsilon>)}{A_M A' \Delta P_C} \right) \left( \frac{1}{K_o \left[ \frac{\partial P}{\partial \xi} \right]_o} \right) V$$

$$G = \frac{\rho_L s}{\rho_s (1 - <\varepsilon>) (1 - s) - \rho_L <\varepsilon> s}$$



# Separation Performance

*Primary Physical Issues to Resolve in Selecting and Specifying Equipment for a Particular Solid-Liquid Separation*

- Mechanism: Filtration or Sedimentation – Is choice critical?
  - Effective, practical filter medium identified?
- Capacity for Liquid Throughput (availability and cost of functional separation area)
  - Pre-Concentration of Feed Suspension?
  - Filter Aid Body Feed?
  - Chemical Pre-Treatment of Feed Suspension? Coagulation and/or Flocculation?
- Capacity for Solids Throughput
- Quality of Liquid Produced
  - Filter Air Precoat?
- Quality of Solids Produced
  - Volatiles Content (Need for deliquoring)
  - Purity (Need for solvent washing)
- Scale of Operation Suited to Technology
- Comparative Performance of Staged Operations

# Duty Specification

*Primary Practical Issues to Resolve in Selecting and Specifying Equipment for a Particular Solid-Liquid Separation*

- Process Integration
  - Continuous or Batch Operating Mode? Is choice critical to contingent process steps?
  - Requirements for Further Processing of Separated Phases? Solids handling? Filtrate polishing?
  - Operating Temperature?
- Disposition of “Waste” Products or By-Products
- Process Containment
  - Environmental, Health and Safety Requirements: Flammability? Toxicity?
  - Sterility or Other Isolation Required?
- Materials of Construction
  - pH adjustment? Abrasivity of Solids? Operating temperature?
- Space (footprint, overhead)
- Practical Scale (S/N #1?) and Complexity of Operation
- Availability and Skill of Operating Resources
- Capital and Installed Costs vs. Budgeted Allocation
- Recurring Maintenance Costs
- Comparative Reliability and Cost of Staged Operations



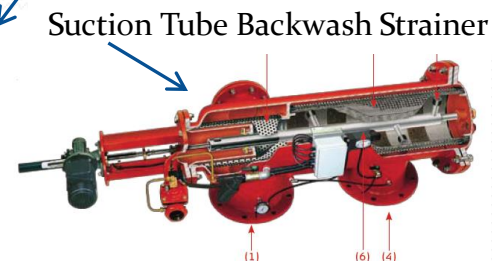
# Pressure Filtration Equipment

- Self-Cleaning Strainers (scraped surface, backwash)

- Screen openings practically limited to  $\geq 20$  microns
- Limits on feed solids concentration
  - Backwash strainers practically limited to 500 ppm because of accumulation; optimum performance  $< 200$  ppm.
  - Scraped surface strainers can remove 5000 ppm at aperture size; can classify concentrated suspensions, remove gels.
- Feed viscosity tolerance
  - Backwash: low ( $< 10$  cP)
  - Scraped Surface: high ( $< 1000$  cP)



Scraped Surface Strainer

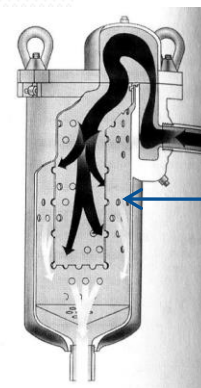
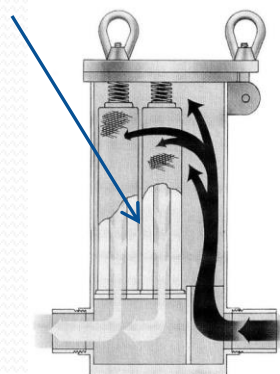


Suction Tube Backwash Strainer

- Standard Filter Bags and Filter Cartridges

- Typical effective filter areas:
  - Size 2 filter bag: 4.4 ft<sup>2</sup>
  - 30" cartridges: 1.64 ft<sup>2</sup> cylindrical, 13.5 ft<sup>2</sup> pleated
- Optimum solids load:
  - Size 2 filter bag: 3.63 kg,  $\leq 400$  ppm feed
  - 30" cartridge: 0.21 kg cyl., 0.51 kg pleated,  $\leq 40$  ppm feed

Filter Cartridges

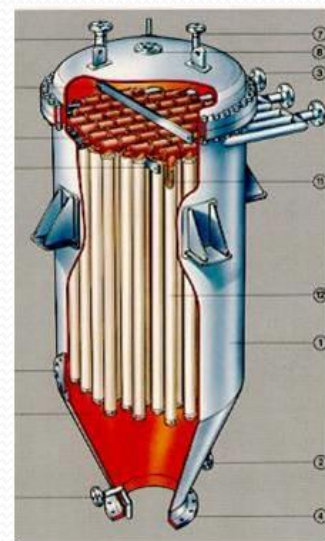


Filter Bag

- Backpulse Candle Filter

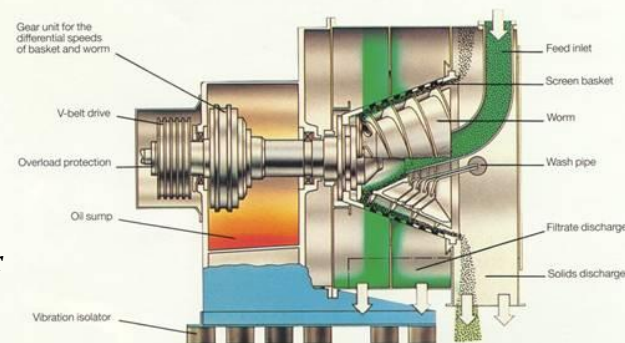
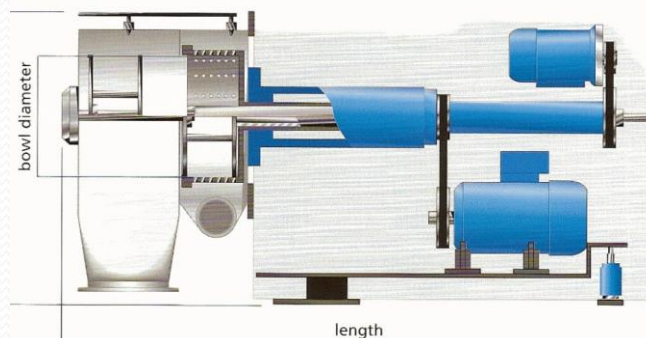
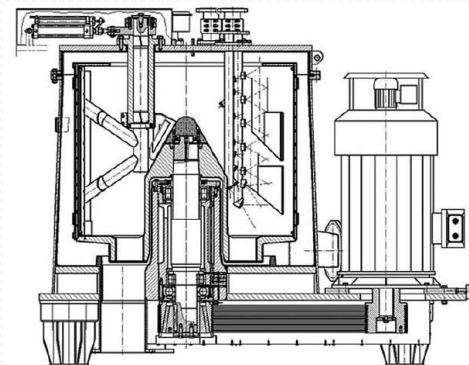
- Highly versatile filter design with large area: capable of producing dry cake, washing solids, using precoat, concentrating suspensions.
- Applicable concentrations
  - Feed suspension solids:  $< 5$  wt% "convenient",  $< 10$  wt% practical
  - Filter cake for dry discharge: 40 – 75 wt% solids
  - Thickened slurry discharge: 10 – 30 wt% solids

DrM Fundabac/Contibac  
Backpulse Candle Filter



# Centrifugal Filtration Equipment

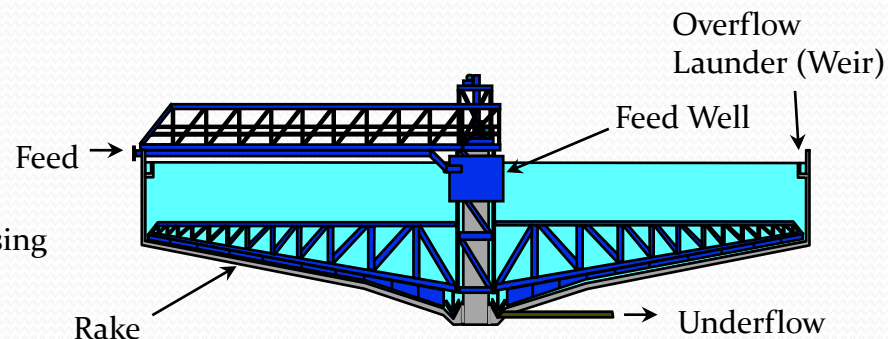
- **Vertical Basket Centrifuge (VBC)**
  - Versatile, fully enclosed, proven history of processing diverse specialty chemicals.
  - Relatively large filter areas available.
  - Very dry cakes:  $\leq 85$  wt% solids aq.,  $\leq 99.5$  wt% volatile solvent
  - Moderately efficient displacement washing.
  - Balance & vibration can present problems.
  - Moderately expensive.
- **Inverting Basket Centrifuge (horizontal basket)**
  - Extremely versatile, thin cakes practical. Very dry cakes similar to VBC.
  - Pressurized gas-assist possible to aid filtration and cake deliquoring, inert head space.
  - Superior washing with horizontal deposit.
  - Relatively small bowls and filtering surface, frequent cycles.
  - Very expensive, complex electronic controls using load cells.
  - Cloths require roughly biweekly replacement, fairly simple procedure.
- **Scroll Screen (Worm Screen) Centrifuge**
  - Relatively inexpensive, smaller machines with superior unit capacity.
  - Smaller screen openings ( $\geq 40 \mu$ ) can be used vs. pusher style centrifuge.
  - One-stage washing of solids possible after initial separation.
  - Higher solids content in filtrate (no cake formed).
  - Discharged solids will contain a higher liquid content compared to other filtering centrifuges.



# Sedimentation Equipment

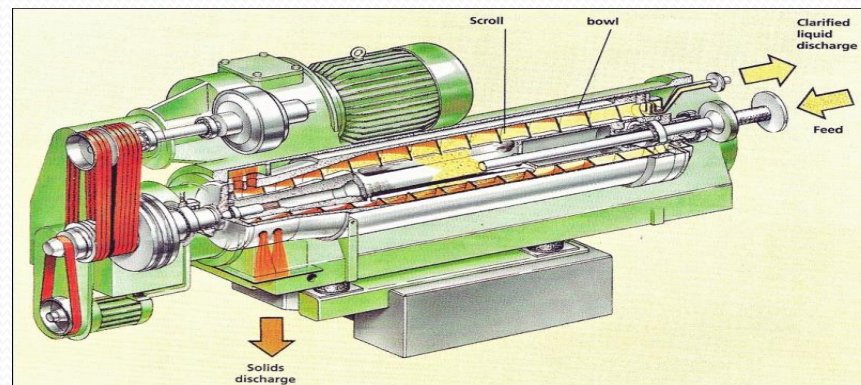
## • Conventional Circular Gravity Settler

- Moderate in cost, simple to operate
- Requires substantial space for often large footprint
- Underflow high in liquid content requires additional processing
- Typically requires chemical pretreatment system; can deliver good overflow clarity but limit on smallest particle captured.



## • Solid Bowl Scroll Decanter Centrifuge

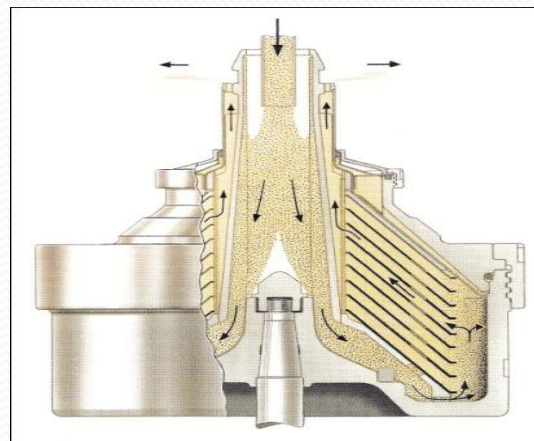
- Highly versatile device in terms of feed and performance
- High throughput capacity for deliquoring
- Sediment moisture can be fair to very good
- Moderate overflow clarity: ~ 300 – 1000 ppm TSS
- High Initial cost; regular skilled maintenance required.



## • Disk Stack Centrifuge

- Excellent clarifying device: < 100 ppm TSS when optimized
- Good throughput capacity in compact machine.
- Feed solids concentration limited to 8 wt% or less
- Wet sediment discharged as paste or thick slurry.
- Capable of separating 3 phases.
- High initial cost and regular skilled maintenance required.

Clarifier Style





# Solids Deliquoring Equipment

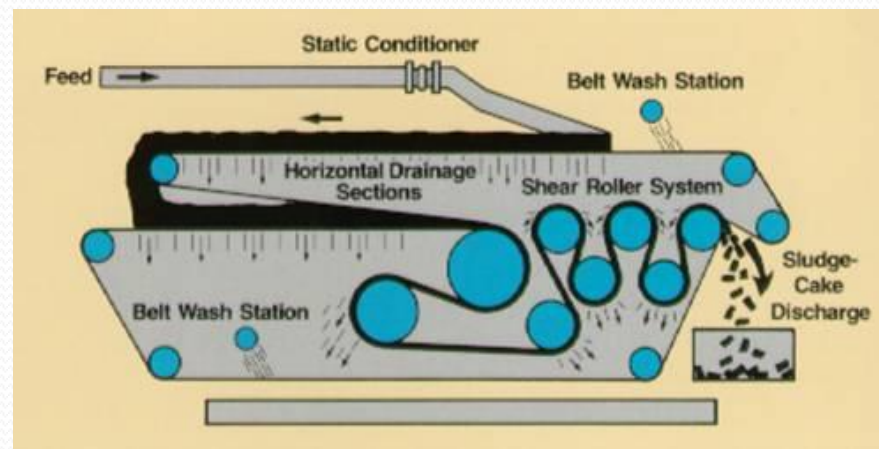
- **Horizontal Chamber Press**

- Highly versatile and effective.
- Batch operated, long cycles, large area possible.
- Can handle very slow filtering solids ( $\geq 0.2$  gpm.ft<sup>2</sup>)
- Can produce cakes of very low moisture:  $\leq 80$  wt% solids, diaphragms improve by 5 – 10 wt%.
- Complete containment or maintaining temperature of process materials is difficult or very complicated.
- Requires some supervision by operating personnel.
- Relatively inexpensive. Conversion to diaphragm plates to shorten cycle and reduce cake moisture adds ~20% to cost.



- **Belt Filter Press**

- Continuous feed to moving serpentine cloth belt.
- Poor containment: only suited to non-hazardous process materials.
- Initial gravity drainage followed by mechanical compression of cake between rollers.
- Requires flocculate/coagulant and/or fibers to obtain cake consistency required to sustain stress without extruding out the open sides.
- Lower solids content in cake compared to chamber or tower press.



# Multipurpose Separations Equipment

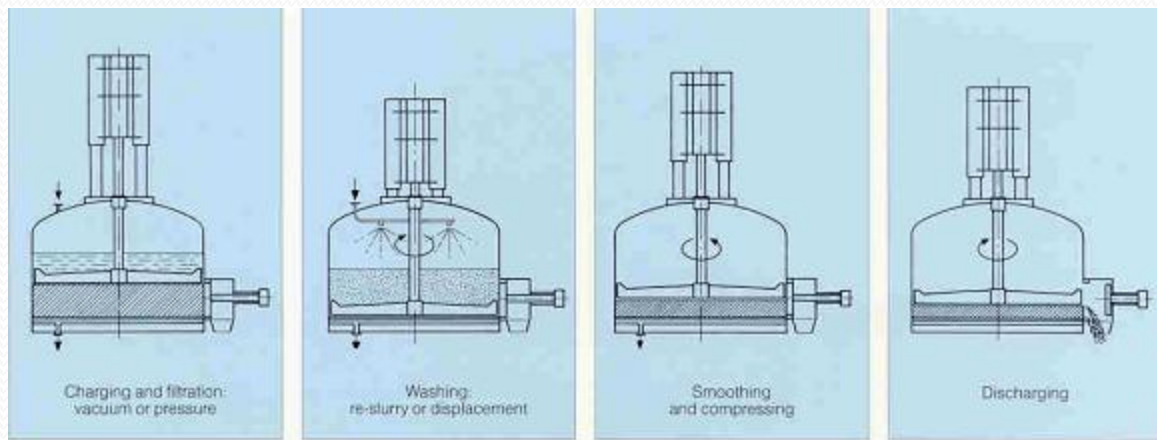
- Outotec Pannevis RT Vacuum Belt Filter

- Solids separation, washing, deliquoring in sequential zones
- Characteristics:
  - Continuous feed with broad turndown.
  - Excellent efficiency for displacement cake washing; potential for countercurrent solvent usage.
  - 3 mm – 50 mm cake depth
  - Cake solids:  $\leq 78$  wt% aqueous,  $\leq 99.5$  wt% volatile solvent
  - No seal belt for improved solvent compatibility



- Nutsche Filter

- Batch-operated, pressure and vacuum capable.
- Accomplishes multiple functions in a single pressure vessel.
- Relatively expensive with limited filtering surface.
- Minimum 4" cake of valuable/difficult solids to be competitive.



# Additional Supporting Material

- Gravimetric Measurement of Solids Concentration in Suspension
- Suspension Rheology
- Nanoparticle Optical Size Measurement (DLS)
- Common Gravity Sedimentation Test Procedures
- Improved Application of Sigma Method
- Staged Application of Hydrocyclones
- References for Solid-Liquid Separations



# Gravimetric Measurement of Solids Concentration in Suspension

- The concentration of insoluble solids in feed, filtrate, decanted liquid, dilute suspensions or slurries is directly determined by weighing.
  - Measure tare weight of a clean, dry filter medium having well defined, high removal efficiency (i.e. “absolute” at size)
    - Whatman glass fiber media grades 934-AH (1.5  $\mu$ ), GF/B (1.0  $\mu$ ), GF/F (0.7  $\mu$ ), or equivalent.
    - 0.1, 0.2, 0.45-micron membranes (PES, nylon, PP, PVDF, PTFE, cellulose acetate).
    - Choose a diameter that will allow all solids to be captured with < 2 mm deep deposit.
  - Filter a net weight of well-mixed sample, rinsing with enough clean solvent to include all solids in containers and displace soluble residue from deposited solids.
  - Dry filter medium with captured solids inside oven protected from air currents at controlled, compatible temperature to completely evaporate solvent without losing or decomposing solids.
  - Weigh the net dry solids captured then divide by the net weight of starting sample to calculate the original solids concentration. The result is termed “total suspended solids” and abbreviated TSS.
- Concentrated slurries, sediment or sludge and filter cakes whose saturating liquid contains negligible soluble species can be simply weighed, dried and weighed again to determine “loss on drying” or moisture.
  - A programmed moisture balance conveniently accomplishes this procedure for quantities less than 10 g.
  - Displacement washing of deep deposits to remove soluble species is generally inaccurate due to maldistribution of wash solvent. Resuspension washing yields a more accurate result for high concentrations of solubles.
  - Solid deposits that tend to crack cannot be effectively washed to remove soluble species in interstitial solution.
- Difficult-to-filter solids can be isolated by centrifuging suspension in a bottle, decanting solids-free liquid, resuspending solids in clean solvent, mixing to extract solubles, repeating before final drying.
- Solid weight-fraction ( $s$ ) is converted to solid volume-fraction ( $\epsilon_s$ ) by a material balance:

$$\epsilon_s = \frac{\frac{s}{\rho_s}}{\frac{s}{\rho_s} + \frac{(1-s)}{\rho_L}} = \left[ 1 + \frac{\rho_s(1-s)}{s\rho_L} \right]^{-1} \quad \begin{array}{l} \rho_s = \text{intrinsic density of solid phase} \\ \rho_L = \text{liquid phase density} \end{array}$$

# Suspension Rheology

- To design and specify the piping that carries feed suspension destined for separation, an estimate of its effective viscosity is often required. Slurry viscosity can be described as a dimensionless factor  $\mu_R$  of the viscosity of transporting liquid  $\mu_L$  :

$$\mu_S = \mu_R \bullet \mu_L$$

Several models describe the relative viscosity as a function of the volume fraction of solid particles  $\phi$ . The choice of model depends on the size and concentration of solids.

- For very low concentrations of fine particles, Einstein's equation (1906) applies:

$$\mu_R = 1 + 2.5 \bullet \phi$$

- For higher solids concentrations, a modified equation proposed by Guth and Simha (1936) takes into account the interactions between solid particles:

$$\mu_R = 1 + 2.5 \bullet \phi + 14.1 \bullet \phi^2$$

This equation was further modified by Thomas (1965) based on fitting experimental data.

$$\mu_R = 1 + 2.5 \bullet \phi + 10.05 \bullet \phi^2 + A \bullet e^{B \bullet \phi}$$

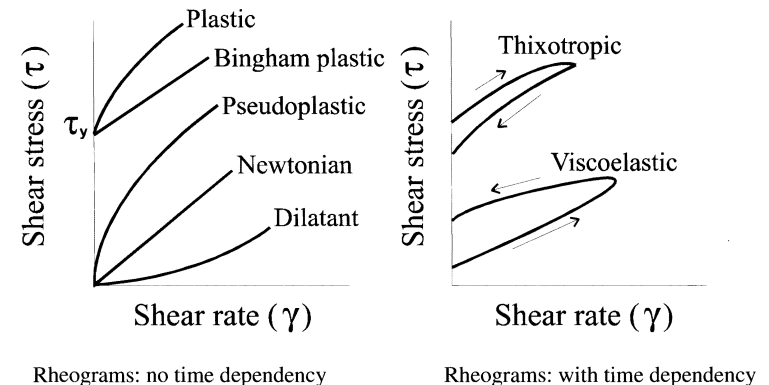
where  $A = 0.00273$  and  $B = 16.6$ .

- For very high solids concentrations, another empirical equation was proposed (1981) by Kitano, Kataoka and Shirota:

$$\mu_R = \left(1 - \frac{\phi}{A}\right)^{-2}$$

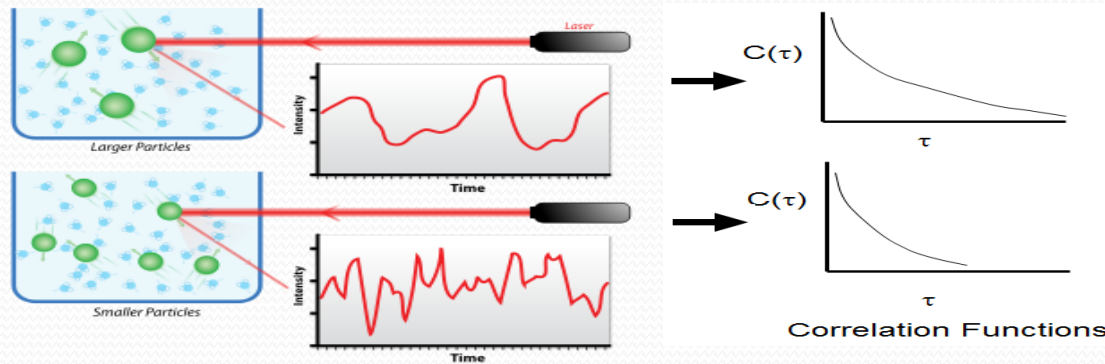
where  $A = 0.68$  for smooth spherical particles.

- Many solid-liquid suspensions display pseudoplastic behavior.
- Thixotropic filter cakes are not uncommon and complicate transport.



# Nanoparticle Optical Size Measurement

- Ensemble Techniques (cont.)
  - Dynamic Light Scattering (DLS)
    - Also called quasi-elastic light scattering or photon correlation spectroscopy
    - Useful size range: 5 nm to several microns, esp. less than 300 nm
    - Characteristic particle dimension vs. wavelength of incident light beam
      - Larger → Mie scattering region → Particle volume from scattered light intensity
      - Smaller → Rayleigh scattering → Time behavior of fluctuations in light intensity



- Key Concept: time scale of fluctuations in scattered light intensity depends on the size of particles.
  - Each individual scattered wave detected by photomultiplier tube (PMT) bears a phase relationship with the incident laser wave that depends on the exact position of originating particle. Scattered intensity is the result of superimposed individual scattered waves (interference).
  - Particles move (diffuse) in random-walk Brownian motion where speed depends on temperature. Small changes in position generate significant changes in phase.
  - Autocorrelation Function relates incident wavelength and scattered wave phase to particle movement and therefore size. Correlation decays exponentially with time.

For a monodispersed suspension:  
where

$$C(\tau) = B[1 + A \exp(-2\Gamma\tau)]$$

Linewidth:

$$\Gamma = \mathcal{D}q^2$$

$$q = \frac{4\pi n_o}{\lambda_o} \sin\left(\frac{\theta}{2}\right)$$

Scattering wave vector:

$$\mathcal{D} = \frac{\kappa T}{3\pi\eta d_H}$$

Stokes-Einstein equation:

$\mathcal{D}$  – Diffusivity  
 $\kappa$  – Boltzman's constant  
 $T$  – Absolute temperature  
 $\eta$  – Suspending liquid viscosity  
 $d_H$  – Hydrodynamic diameter

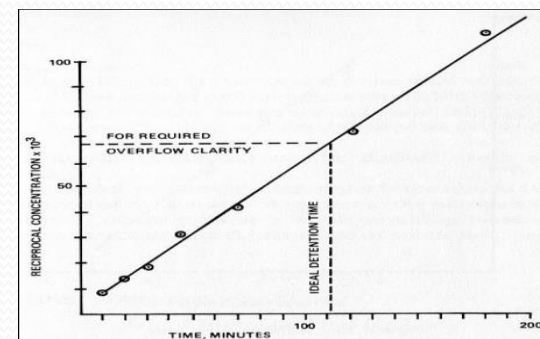
# Common Gravity Sedimentation Test Methods

- Short Tube Procedure

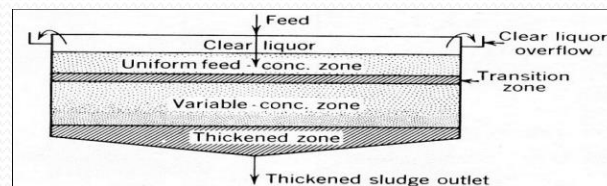
- Overflow solids concentration depends on overflow rate alone.
- Valid for particulate settling only.
- Steps:
  - Decant and measure TSS and PSD's for various times. Overflow rate determined from height of short tube and the time to achieve required removal efficiency at specified size and efficiency.
  - Calculate area for required volumetric throughput. Apply area efficiency factor.

- Second Order Procedure (Also called “Bulk Settling Test”)

- Overflow solids concentration essentially determined by detention time with little dependence on overflow rate.
- Valid for flocculated (zone) settling only.
- Steps:
  - Measure solids concentration in supernatant liquid for a series of retention times. Plot reciprocal concentration vs. detention time (second-order plot).
  - Determine ideal retention time for required overflow clarity using linear dependence of reciprocal dimensionless overflow concentration on detention time (concentration at infinite time small). Apply detention efficiency factor (~50%).
  - Determine bulk settling rate (interface vs. time) then set equal to maximum overflow rate after dividing by design factor of 2 (50% area efficiency).
  - Calculate clarifier height from overflow rate and detention time. Calculate area that satisfies specified volumetric throughput and measured overflow rate.
    - Note: If settling rate becomes excessively low ( $< 0.1$  cm/s) or interface does not develop, modify feed slurry with coagulant or flocculant and repeat test.



Second Order Plot



Zone Settling in Continuous Clarifier

## Improved Application of Sigma Method

- Definition and Experimental Determination of  $\Sigma$

- Major shortcoming of the  $\Sigma$  concept and method is that cut size is an insufficient criterion for separation efficiency. Different efficiencies can be obtained for a given cut size depending on the size distribution of feed solids (e.g. different PSD's can have the same median size).
- Grade efficiency  $G(x)$  is contained in the general expression for  $\Sigma$  and could be determined from experimental test results (sediment PSD) although there is ample opportunity for error in that measurement.
- More accurate and convenient to measure the fraction of unsedimented solids,  $(1 - E_T)$  where

$$E_T = \frac{x_c(1 - x_o)}{x_o(1 - x_c)}$$

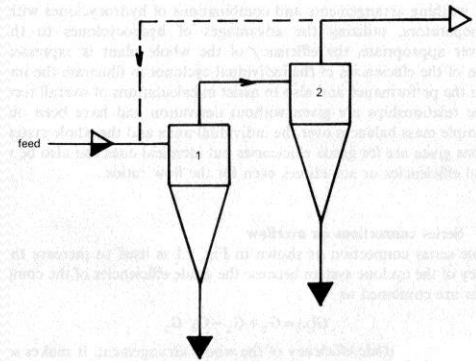
and  $x_o$  equals the mother liquor concentration,  $x_c$  equals the concentration in overflow centrate.

- Plot the fraction solids not captured  $(1 - E_T)$  against the ratio of measured flow rate to calculated  $\Sigma$  value. Vary  $\Sigma$  by changing the speed of rotation. Curve is often a straight line on log-probability coordinates. Although that correlation is a function of the feed particle size distribution, the ratio of  $Q/\Sigma$  for a specified overall removal efficiency for that suspension can be located.

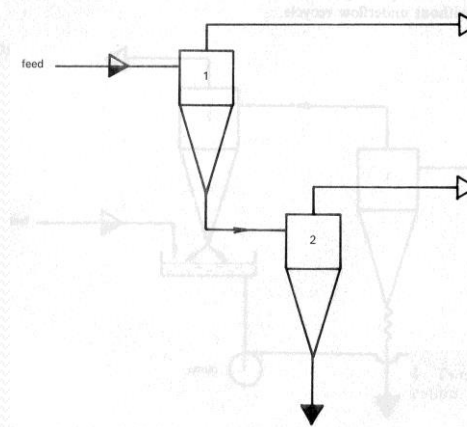


# Staged Application of Hydrocyclones

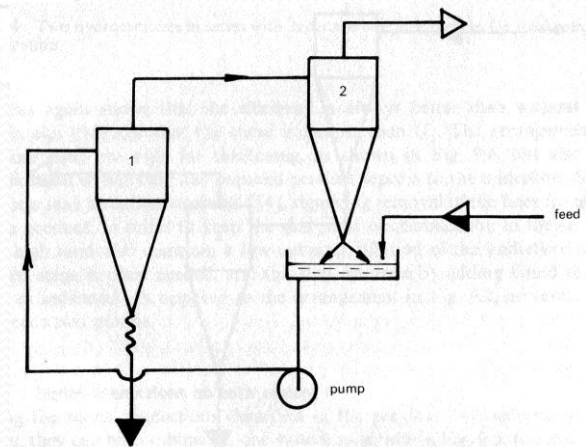
- Clarification



- Slurry Thickening



- Combined Clarification and Thickening





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