# Separation Processes

## ChE 4M3



kevin.dunn@mcmaster.ca
http://learnche.mcmaster.ca/4M3

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# Administrative

Assignment 2 is posted

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- e.g. `4M3-Sivakumaran-Dunn-Assignment-2`

# Some context

The three most important characteristics of an individual solid particle are its composition, its size and its shape.

Composition: affects density, conductivity, *etc*, if the particle is uniform

# Particle shape characterization

A particle may be regular shaped: *spherical* or *cubic*

- capable of precise definition using equations

Irregular shaped: e.g. broken glass, sand, rock, most solids

- properties of irregular shapes are expressed ito a regular shaped particle's characteristics

# Particle shape: Sphericity

Why use a sphere?

- it has the same shape from all angles
- behaves the same way from all angles

Other particles behave less ideally; we define sphericity as one metric of a particle's shape:

$$\psi = \frac{\text{surface area of sphere with same volume as particle}}{\text{surface area of particle}}$$

For all particles: $0 < \psi \leq 1$

To try: calculate the sphericity of a cube with side length $= c$

Answer: $\psi = 0.806$

# Other shape metrics

Find the diameter of a sphere that has the same ______ as the particle

- volume
- surface area
- surface area per unit volume
- area in the direction of travel [drag diameter]
- projected area, but in a position of maximum stability
- settling velocity [Stokes' diameter]
- will fit through the same size square aperture [sieve diameter]

# Particle size characterization

So far we have assumed particles to be separated are of a single size. This is never true: there is always a size distribution.

Particle size: affects surface per unit volume, rate of settling in a fluid, *etc*

## Aims

How do we measure this distribution?

How do we describe (characterize) a size distribution?

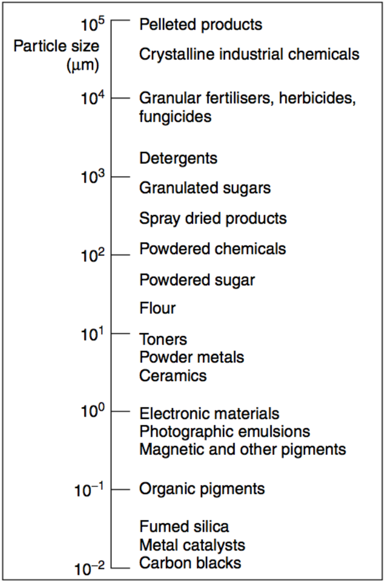
- what is the "**average**" particle size?

# Other reasons to consider particle size distributions?

- Understand your colleagues: "After crushing, the feed may be ground in several stages from a size of 5 to 6 cm to a powder of 75 to 90 percent passing a 200-mesh sieve."
- Solid material handling industry (foods, grains, pulp, sand, cement, coal, *etc*): we must deal with distributions
- e.g. activity of a powdered drug $= f$(particle size)
- e.g. "hiding power" of a paint/pigment $= f$(particle size)

We will require this understanding for future sections: filtration, flow of fluids through packed beds, membranes, and so on.

# Some typical particle sizes




[Richardson and Harker, 5ed, p4]

# Standard screens


Mesh 10 screen = 2.00 mm opening = 10 openings per linear inch.

# Standard screen sizes

The US standard (Tyler series). Selected examples are:

| Mesh number | Square aperture opening ($\mu$m) |
|---|---|
| 3.5 | 5600 |
| $\vdots$ | $\vdots$ |
| 10 | 2000 |
| $\vdots$ | $\vdots$ |
| 20 | 850 |
| 25 | 710 |
| 30 | 600 |
| $\vdots$ | $\vdots$ |
| 140 | 106 |
| 170 | 90 |
| 200 | 75 |
| 230 | 63 |
| $\vdots$ | $\vdots$ |
| 450 | 32 |

Tyler standard:

- e.g. 75$\mu$m opening: called 200 mesh screen
- i.e. apertures per inch = 200 mesh screen

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- Successive apertures decrease by factor of $\sim \sqrt[4]{2}$

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- Other standards: British I.M.M. and U.S. A.S.T.M.

# Screens

Stack screens: apertures from largest on top to smallest


[http://en.wikipedia.org/wiki/File:Laborsiebmaschine_BMK.jpg]

# Screens

- Top screen usually has 100% material passing through
- Shaken for a predetermined time; or rate of screening levels out
- Shake intensity balanced: not too aggressive to break particles apart
- Smaller particles tend to stick to each other, so small size fractions inaccurate
- Done on wet or dry material
- Wet screening: washes smaller particles off larger ones

# Other particle size methods

- Sedimentation: pipette or an immersed scale
- Elutriation: reverse sedimentation

$$v_{\text{TSV}} = \frac{(\rho_p - \rho_f)\, g D_p^2}{18\mu_f}$$

$$D_p = \sqrt{\frac{18\mu_f\, v_{\text{upward}}}{(\rho_p - \rho_f)\, g}}$$

- Permeability methods
  - flow $= k\Delta P$; where $k \propto \dfrac{\text{surface area}}{\text{volume}}$
  - from which we can calculate equivalent spherical diameter
- Laser diffraction
  - uses principle of radial laser beam scattering
  - handles ranges from 0.1 to 600 $\mu$m
  - can be applied online for real-time monitoring

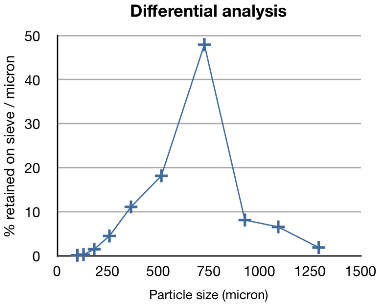
Many others: see Perry's Chapter 21

# Example

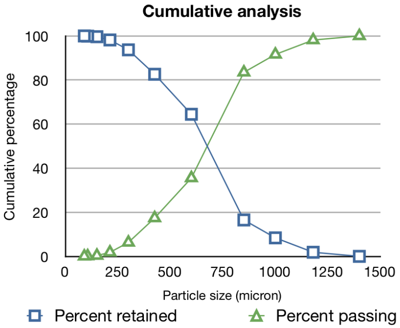
| Mesh | Aperture [$\mu$m] | Mass retained [g] | Avg size* | Cuml. % passing |
|---|---|---|---|---|
| 14 | 1400 | 0 | - | 100 |
| 16 | 1180 | 9.1 | 1290 | 98.1 |
| 18 | 1000 | 32.1 | 1090 | 91.6 |
| 20 | 850 | 39.8 | 925 | 83.5 |
| 30 | 600 | 235.4 | 725 | 35.5 |
| 40 | 425 | 89.1 | 513 | 17.4 |
| 50 | 300 | 54.4 | 363 | 6.3 |
| 70 | 212 | 22.0 | 256 | 1.8 |
| 100 | 150 | 7.2 | 181 | 0.4 |
| 140 | 106 | 1.2 | 128 | 0.1 |
| Pan | 0 | 0.5 | 53 | 0.0 |
| Sum | | **491** | | |

$*$ *average screen size used for differential plots*

# Differential and Cumulative analysis




*Plot using average screen sizes*




*Plot using actual screen sizes*

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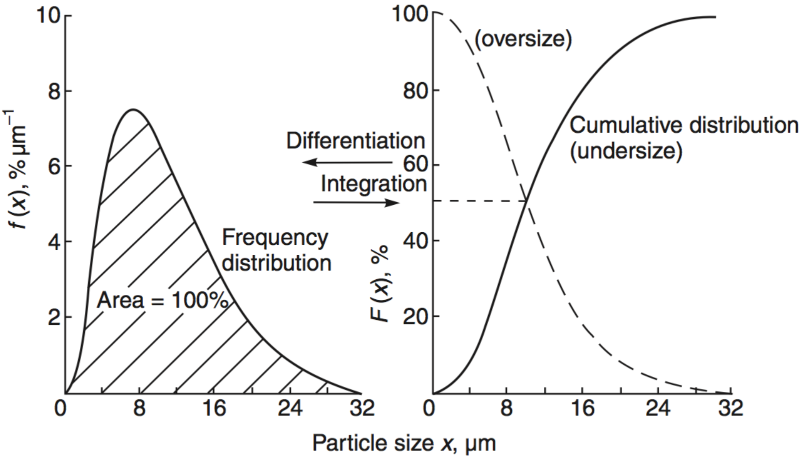
Theory:

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

$F(x) =$ percent passing curve

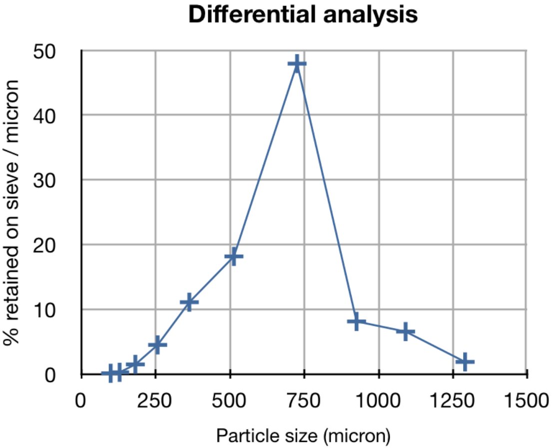
# Theoretical view




[Svarovsky, 4ed, p42]

# Mean diameter calculations

A number of mean diameters can be calculated. These can be derived from the cumulative analysis plot:



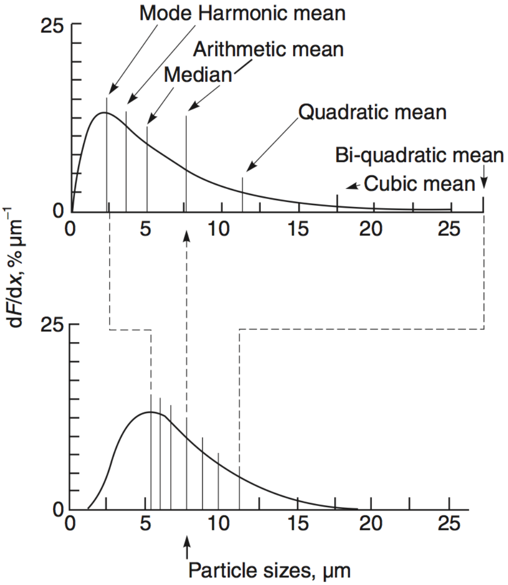

- Arithmetic mean = 318 $\mu$m
- Volume mean diameter = 430 $\mu$m
- Surface mean diameter (Sauter mean diameter) = 565 $\mu$m
- Weight or mass-mean diameter = 666 $\mu$m

- Seader, 3ed, p 678 - 679
- Svarovsky, 4ed, p37 - 43

# Which mean should I use?

- Rather use the distribution curve, if available
- If one has to resort to a single number, use what is appropriate
  - volume mean diameter: used for packing estimation
  - surface mean diameter: used for skin friction, and mass transfer calculations
- The idea is that if two materials had the same “mean diameter”, that they would behave the same way in the application being considered.

# Two distributions, same arithmetic mean




[Svarovsky, 4ed, p59]

# Sampling a stream

Particle size measurements are strongly dependent on the sample taken. The "golden" rules of sampling:

1. take sample from a moving stream: dry powders and slurry
2. sample *whole* stream for many short periods (not part of stream for whole time)

There are books written on the topic of sampling. Consult an experienced person if important decisions rest on the sample taken.

# Main references


1. Richardson and Harker, “Chemical Engineering, Volume 2”, 5th edition, Chapter 1
2. Perry’s Chemical Engineers’ Handbook, Chapter 21.1
3. Seader, Henley and Roper, “Separation Process Principles”, page 675 to 679 in 3rd edition

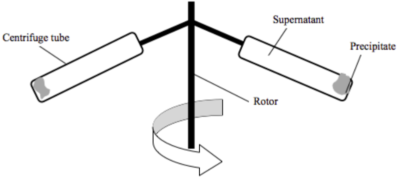
# Why consider centrifuges?

- When gravity (freely available) is not fast enough
- Decrease the separation time and increase *separation factor*
- Much smaller piece of equipment
- Achieve separations not possible by gravity:
  - overcome Brownian limits
  - overcome convection currents
  - overcome stabilizing forces that hold an emulsion together

Why not just apply flocculation?

# Terminology


[http://en.wikipedia.org/wiki/File:Tabletop_centrifuge.jpg]




- **Suspension**: the mixed material added into the centrifuge tube
- **Pellet** or **precipitate**: hard-packed concentration of particles after centrifugation
- **Supernatant**: clarified liquid above the precipitate

# Uses

Used since 1700's:

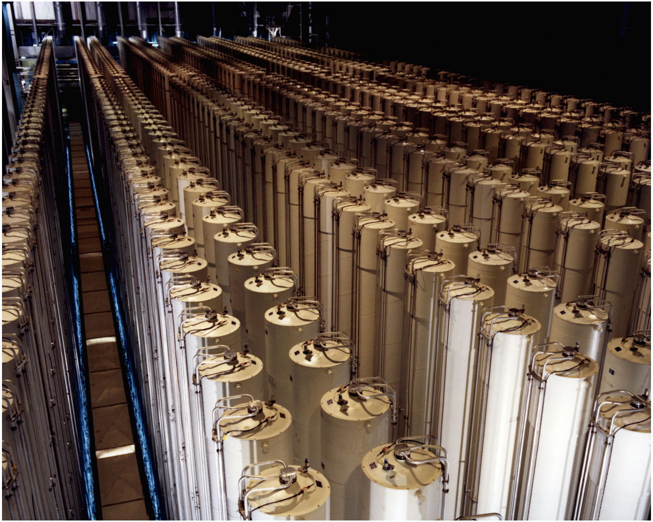
1. separate particles from fluid based on density
2. separate immiscible fluids (liquid and gas!) of different densities
3. to enhance drainage of fluid from particles for drying
4. enhance mass transfer (look at centrifugal packed bed contactors in your own time)

Examples:

- Cream from milk (milk is an emulsion)
- Clarification: juice, beer (yeast removal), essential oils
- Widely used in bioseparations: blood, viruses, proteins
- Remove sand and water from heavy oils

# Interesting use: gas-gas separation

- Uranium enrichment in a Zippe-type centrifuge: U-235 is only 1.26% less dense than U-238: requires counter-current cascade



[http://en.wikipedia.org/wiki/File:Gas_centrifuge_cascade.jpg]

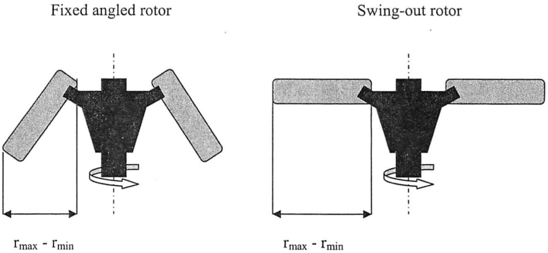
# Principle of operation

- items being separated must have a density difference
  - Video
- centrifugal force acts outward direction $= ma = m(r\omega^2)$
  - $m =$ particle's mass [kg]
  - $r =$ radial distance [m]
  - $\omega =$ angular velocity [rad.s$^{-1}$]
  - recall $2\pi$ rad.s$^{-1}$ $=$ 1Hz
  - and 1 rad.s$^{-1}$ $\approx$ 9.55 revolutions per minute
  - $G = \dfrac{mr\omega^2}{mg} = \dfrac{r\omega^2}{g}$

| **Example** | **Revolutions per minute** | **G's** |
|---|---|---|
| Car going round and round | 10 to 15 | 1 to 2 |
| Washing machine at home | 1500 | 625 (r=0.25m) |
| Industrial centrifuge | $<$ 15000 | 25000 (r=0.1m) |
| Laboratory centrifuge | 30,000 to 100,000 | 100,000 to 800,000 |
| Zippe-type centrifuge* | 90,000 | $\sim 1 \times 10^6$ |

* tangential velocity $>$ Mach 2 $\sim$ 700m.s$^{-1}$

# Laboratory centrifuges




Main selection factors:

1. duration $= t$ [use minutes in the equation below]
2. maximum rotational speed $= \mathrm{RPM}_{\mathrm{max}}$

$$t = \frac{k}{S}$$

- $S =$ Svedberg coefficient of the material (from tables, experiments)
- $r_{\mathrm{max}}$ and $r_{\mathrm{min}}$ as shown above [cm]
- $k = 2.53 \times 10^{11} \left( \frac{\ln\left(r_{\mathrm{max}} - r_{\mathrm{min}}\right)}{\mathrm{RPM}_{\mathrm{max}}^2} \right)$

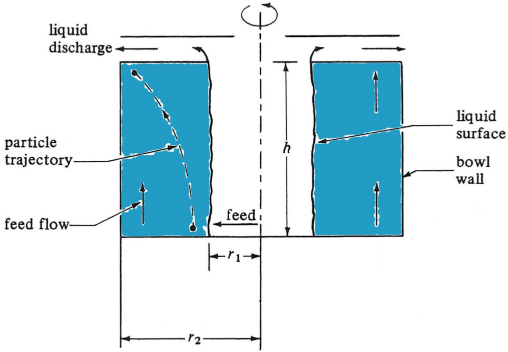
e.g. $S_{20} = 6.43$ for collagen

# Theoretical trajectories: tubular bowl centrifuge

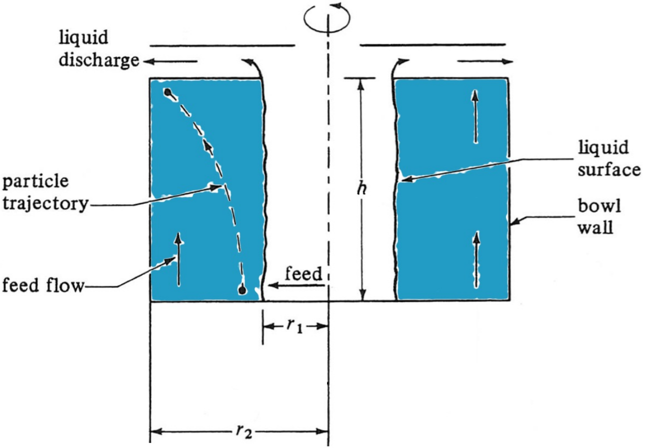
- Most commonly used for small particle separation
- Stokes' law applies

$$v = \frac{dr}{dt} = \frac{D_p^2 \left(\rho_p - \rho_f\right) r\omega^2}{18\mu_f}$$

- The particle is also forced in the direction of fluid flow, so its net trajectory:



# Theoretical trajectories: tubular bowl centrifuge




Integrate from $t = 0$ where $r = r_1$ to the outlet, where we require the particle to be exactly at $r = r_2$ within a time of $t = t_T$ seconds:

$$t_T = \frac{18\mu_f}{D_p^2\left(\rho_p - \rho_f\right)\omega^2} \ln \frac{r_2}{r_1}$$

# Theoretical trajectories: tubular bowl centrifuge

So $t = t_T$ represents the minimum time the particle should spend in the centrifuge: minimal residence time.

The volume of fluid in the centrifuge is $V = \pi \left(r_2^2 - r_1^2\right) h$.

Calculate the maximum volumetric flow rate, $Q_{\max}$ [m$^3$.s$^{-1}$]

$$Q_{\max} = \frac{V}{t_T} = \frac{D_p^2 \left(\rho_p - \rho_f\right) \omega^2}{18\mu_f \ln(r_2/r_1)} \pi \left(r_2^2 - r_1^2\right) h$$

- Alternative interpretation: for a given flow $Q$, find the largest particle diameter that will arrive exactly at $r_2$ at height $h$
- Assume particles with smaller $D_p$ leave in supernatant
- Obviously this is excessive: we have a discharge weir to *retain particles* that might not have reached $r_2$ at height $h$

# Cut-size diameter

So to prevent excessive over design, we rather find the halfway mark between $r_1$ and $r_2$, and solve the equations for the diameter of the particle that reaches this *cut point*:

$$Q_{\text{cut}} = \frac{V}{t_{T,\text{cut}}} = \frac{D_{p,\text{cut}}^2 \left(\rho_p - \rho_f\right) \omega^2}{18\mu_f \ln\left[2r_2/(r_1 + r_2)\right]} \pi \left(r_2^2 - r_1^2\right) h$$

- we design for the cut-point volumetric flow rate $Q_{\text{cut}}$
- and solve for the cut point diameter, $D_{p,\text{cut}}$
- all other terms in the equation are known

# Sigma theory for centrifuges

Take the previous equation for $Q_{\text{cut}}$, multiply numerator and denominator by $2g$, then substitute Stokes' law for particles settling under gravity:

$$v = \frac{(\rho_p - \rho_f)\, g D_p^2}{18\mu_f}$$

we obtain:

$$Q_{\text{cut}} = 2\left(\frac{(\rho_p - \rho_f)\, g D_{p,\text{cut}}^2}{18\mu_f}\right) \cdot (\Sigma) = 2v_{\text{TSV}} \cdot \Sigma$$

$$\Sigma = \frac{\omega^2 \left[\pi h \left(r_2^2 - r_1^2\right)\right]}{2g \ln\left[2r_2/(r_1 + r_2)\right]}$$

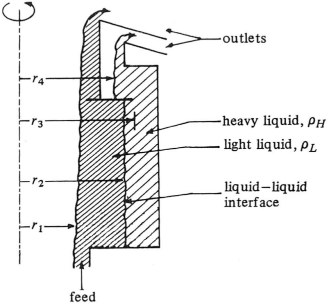
$\Sigma = f(r_1, r_2, h, \omega)$

# Why use the Sigma term?

- $\Sigma = f(r_1, r_2, h, \omega)$
- it is only a function of the centrifuge's characteristics; not the particle or fluid
- $\Sigma$ has units of m$^2$: it is the equivalent surface area required for a gravity sedimentation basin
- Centrifuge A: $Q_{\text{cut},A} = 2v_{\text{TSV}} \cdot \Sigma_A$
- Centrifuge B: $Q_{\text{cut},B} = 2v_{\text{TSV}} \cdot \Sigma_B$
- $$\frac{Q_{\text{cut},A}}{Q_{\text{cut},B}} = \frac{\Sigma_A}{\Sigma_B}$$
- Used for scale-up **of the same feed**, i.e. the same $v_{\text{TSV}}$
- Used for scale-up **within the same types of equipment**
- $\Sigma$ equation is different for other centrifuge types

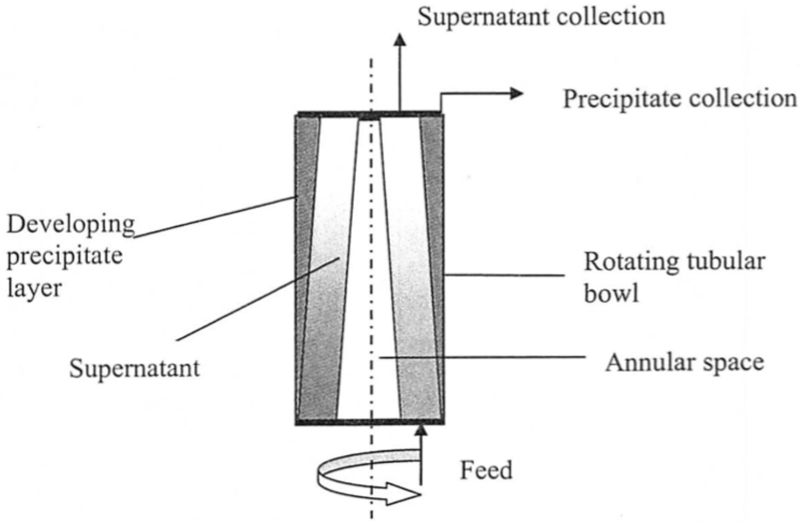
# More on the tubular bowl centrifuge

- Batch operation: stop to clean out solids; restart again; use paper on wall to assist solids removal [$\sim$ 15 min turnaround]
- Contamination possible, *not always* suitable for bioseparations
- A high L/D aspect ratio: stable to operate
- Can be used for fluid-fluid separation




$$\frac{\rho_H}{\rho_L} = \frac{r_2^2 - r_1^2}{r_2^2 - r_4^2}$$

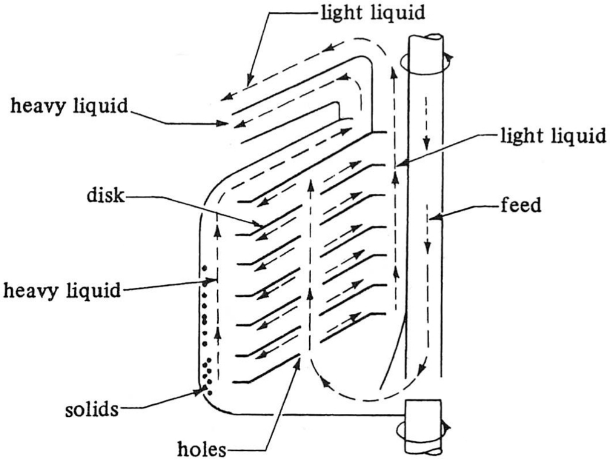
# More on the tubular bowl centrifuge




A diagonal solids layer is built up

[Ghosh, p 84]

# Disk-bowl centrifuges




Video to illustrate operation:
http://www.youtube.com/watch?v=YMbaBLpInrc

Another video: http://www.youtube.com/watch?v=bzXUiLajVlg

# Disk-bowl centrifuges

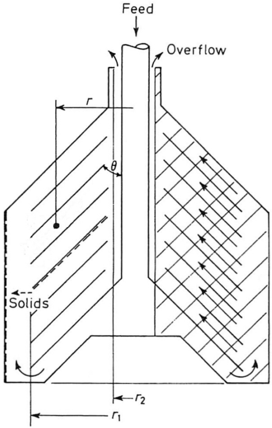
- Recall: $Q = V/t_T$ ($t_T$ will be different for a disk-bowl)
- If we increase volume of fluid, we get higher throughput, $Q$
- Adding angled disks gives a greater surface area, hence volume, without increasing bowl diameter
- Widely used in bioseparations: no contamination (aseptic)
- Also for: fish oil, fruit juice, beverage clarification

# Disk-bowl centrifuges

- Disks angled at 35 to 50°; $\sim$ 50 to 150 disks per unit
- Typically between 0.15 to 1.0m in diameter; with rotational speeds of 0 to 12,000 rpm
- Typically used to treat up to 15% solids in feed stream
- Can be operated continuously (infrequent cleaning of disks)

$$\Sigma = \frac{2\pi\omega^2 N(r_1^3 - r_2^3)}{3g\tan\theta}$$

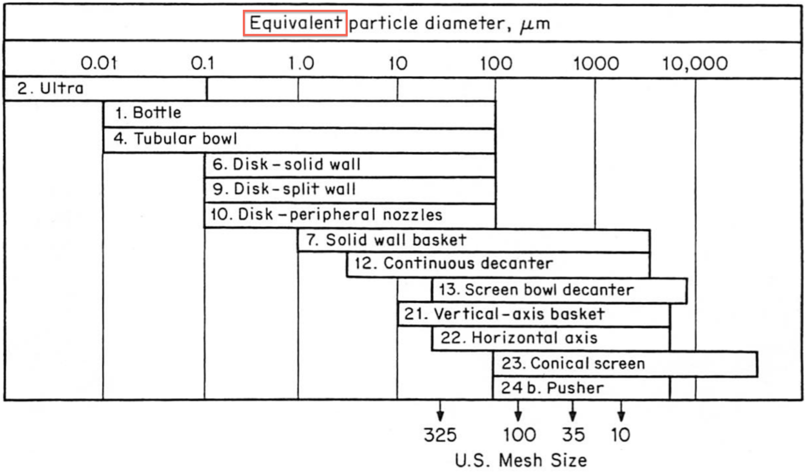
- $N$ = number of disk plates
- $\theta$ = angle of disks
- $r_1$ = outer cone diameter
- $r_2$ = inner cone diameter



# Safety

- careful selection of materials of construction: corrosion and withstand high forces
- heat removal might be required
- rotational equipment requires careful balance
- digital control is critical
  - PLC: programmable logical controllers
  - SCADA: supervisory control and data acquisition
  - safety interlocks
  - cameras are increasingly used to monitor sediment buildup: auto-stop and clean
- flammable fluids (e.g. solvents): nitrogen blanket

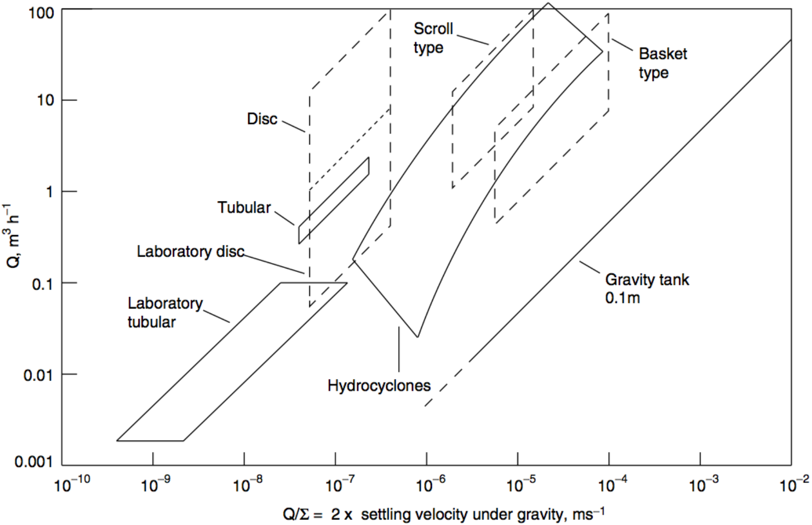
# Choosing a centrifuge unit




[Schweitzer, p 4-58]

# Selecting a centrifuge

Based on required performance



# Design a centrifuge for beer clarification

Design a separation plant to remove suspended yeast cells from beer.

Beer is produced in batches of 100 $m^3$, with 4 batches per day.

Some data:

- Density of beer: 1020 $kg.m^{-3}$
- Density of yeast cells: 1075 $kg.m^{-3}$
- Yeast cell diameters: 4 to 6 $\mu$m
- 11.5 metric tonnes of yeast are suspended in each 100 $m^3$ fermenter
- Asceptic operation is vital

# Example

A lab scale tubular bowl centrifuge has the following characteristics:

- $r_1 = 16.5$ mm
- $r_2 = 22.2$ mm
- bowl height of 115 mm
- 800 revolutions per second

It is being used to separate bacteria from a fermentation broth:

1. If the broth has the following properties:
    - $\rho_f = 1010$ kg.m$^{-3}$
    - $\mu_f = 0.001$ kg.m$^{-1}$.s$^{-1}$
    - $D_{p,\text{min}} = 0.7$ $\mu$m
    - $\rho_p = 1040$ kg.m$^{-3}$

    calculate both $Q_{\text{max}}$ and the more realistic $Q_{\text{cut}}$.
2. Calculate the $\Sigma$ factor for this centrifuge based on the cut-size flowrate.

# References


- Geankoplis, “Transport Processes and Separation Process Principles”, 3rd or 4th edition, Chapter 14
- Richardson and Harker, “Chemical Engineering, Volume 2”, 5th edition, Chapter 9
- Perry’s, “Chemical Engineers’ Handbook”, 8th edition, Chapter 18.8
- Svarovsky, “Solid Liquid Separation”, 3rd or 4th edition. Well written and straightforward to understand.
- Seader et al. “Separation Process Principles”, page 800 to 802 in 3rd edition
- Schweitzer, “Handbook of Separation Techniques for Chemical Engineers”, Chapter 4.5