Separation Processes

ChE 4M3

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- if you let us know about **any errors** in the slides
- **any suggestions to improve the notes**

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or anonymous messages can be sent to Kevin Dunn at

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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.
- Seader et al. "Separation Process Principles", page 800 to 802 in 3rd edition.
- Schweitzer, "Handbook of Separation Techniques for Chemical Engineers", chapter 4.5.

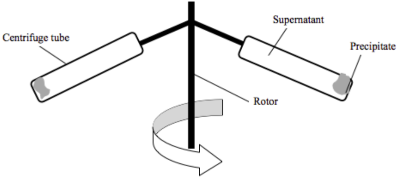
Why consider centrifuges?

- 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:

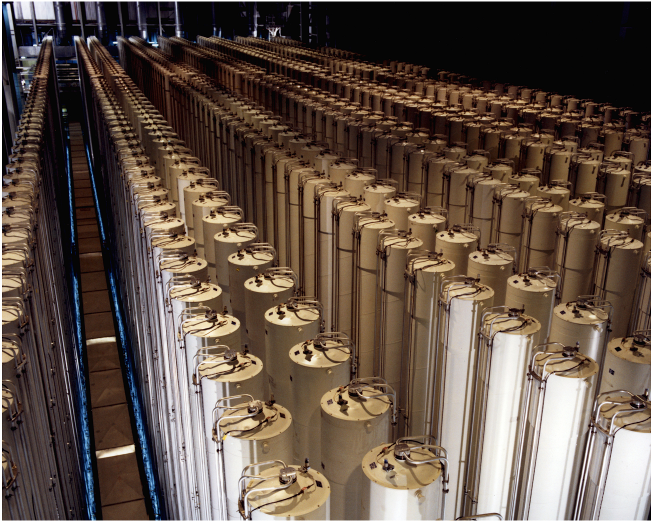
1. separate particles from fluid based on density
2. separates immiscible fluids (liquid and even gases) of different densities
3. to enhance drainage of fluid from particles for drying
4. enhance mass transfer (look at centrifugal packed bed contactors in your own time)

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]

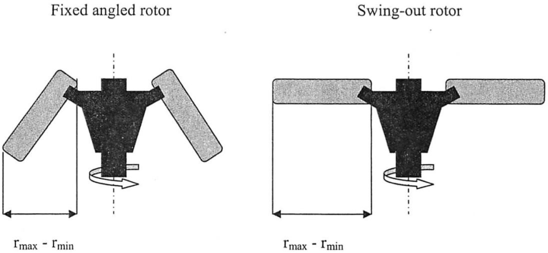
Principle of operation

- items being separated must have a **density difference**
- it is not a difference in the mass, only density
  - Video of emulsion separation at high G's
- centrifugal force acts outward direction $= ma = m(r\omega^2)$
  - $m =$ particle's mass [kg]
  - $r =$ radial distance from center point [m]
  - $\omega =$ angular velocity $= \dfrac{d\theta}{dt}$ [rad.s^{-1}]
  - recall 2π rad.s^{-1} $=$ 1Hz
  - and 1 rad.s^{-1} $\approx$ 9.55 revolutions per minute [rpm]
  - $G = \dfrac{mr\omega^2}{mg} = \dfrac{r\omega^2}{g}$

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 $= \mathrm{RPM}_{\mathrm{max}}$

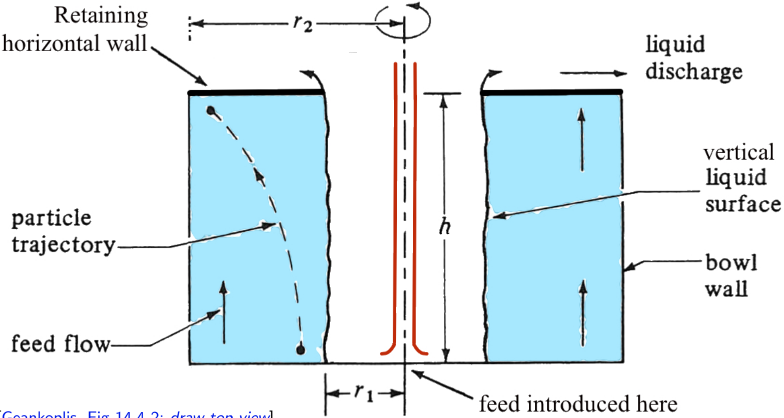
$$t = \frac{k}{S}$$

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

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

Tubular bowl centrifuge

- Most commonly used for small particle separation
- Fluid and suspended solids are fed at the center
- A vertical wall of fluid is formed. Useful video to see this.
- Feed is continually added, forcing fluid out the top, over the retaining wall. Solids accumulate inside the bowl.



[Geankoplis, Fig 14.4-2; *draw top view*]

Recall particles in a fluid: Stokes' law

Let's understand how the solid particles move:

Recall if Re < 1

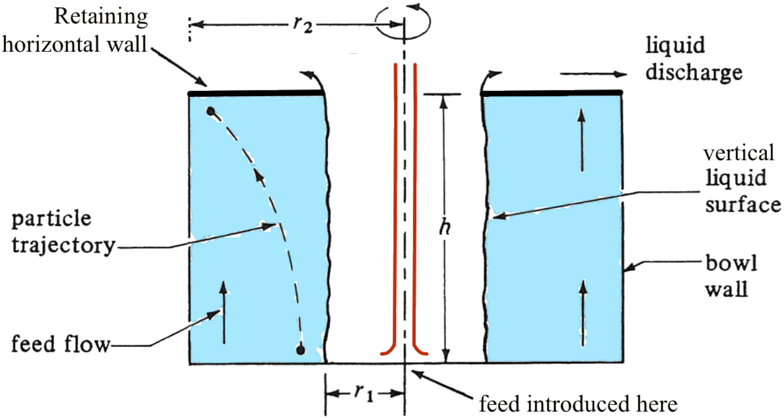
$$v_{\text{TSV}}^{\text{grav}} = \frac{D_p^2\left(\rho_p - \rho_f\right)g}{18\mu_f}$$

In a centrifuge, we have simply replaced g with a centrifugal force, $r\omega^2$ (gravity is negligible)

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

- The particle is also forced in the *vertical* direction of fluid flow at a constant upward velocity, so its net trajectory is curved.
- In centrifuges: particles are likely to have Re < 1 (why?)

Theoretical trajectories: tubular bowl centrifuge



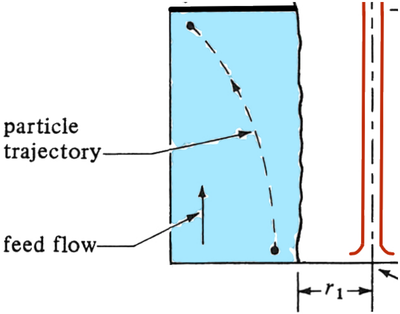
Integrate from $t = 0$ where $r = r_1$ to the outlet, where we require the particle to be exactly at $r = r_2$ within a time of $t = t_*$ seconds:

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

Theoretical trajectories: tubular bowl centrifuge

Consider a particle moving with too slow a horizontal velocity (e.g. centrifuge is too slow).

- Within the time from $t = 0$ to $t = t_*$, this particle is moving too slowly, and will not reach the wall at r_2
- This particle is then assumed to have left in the supernatant (liquid discharge)



$t = t_*$ gives a **bound** on the time it should take a particle to reach the wall at r_2, starting at r_1.

Calculating the centrifuge's throughput, Q

Once we know how long a particle should be in the centrifuge, we can calculate a feed flowrate, Q. The volume of fluid in the centrifuge is $V = \pi\left(r_2^2 - r_1^2\right)h$. Calculate the volumetric flow rate

$$Q_* = \frac{V}{t_*} = \frac{D_p^2\left(\rho_p - \rho_f\right)\omega^2}{18\mu_f \ln(r_2/r_1)}\pi\left(r_2^2 - r_1^2\right)h \qquad [\mathrm{m^3.s^{-1}}]$$

- What happens if we operate a flow rate slower/faster than this Q_*?
- *Alternative interpretation*: for a given flow Q_*, find the largest particle diameter that will arrive exactly at r_2 at height h. Particles with smaller D_p are expected to leave in supernatant.
- Obviously this is excessive: we have the horizontal discharge weir to *retain particles* that might not have reached r_2 at height h
- r_2 remains fixed for a purchased and installed centrifuge (design parameter)

Cut-size diameter

So to prevent excessive over design, we rather find the halfway mark between r_1 and r_2, and solve the same equations to find the time, called t_{cut}, for a particle to reach this cut point:

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

- we design for the cut-point volumetric flow rate Q_{cut}
- and can then solve for the cut point diameter, $D_{p,\text{cut}}$
- all other terms in the equation are known/set
- We can also design for a given diameter, and solve for the Q_{cut}.

Note: We could use any reasonable point between r_1 and r_2. The 50% point is convention. It accounts for uncertainties in our flows, physical properties and idealities assumed with Stokes' law.

Example

A lab scale tubular bowl centrifuge has the following characteristics:

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

It is being used to separate bacteria from a fermentation broth experiment.

If the broth has the following properties:

- $\rho_f = 1010$ kg.m^{-3} ← note how close these are
- $\rho_p = 1040$ kg.m^{-3}
- $\mu_f = 0.001$ kg.m^{-1}.s^{-1}
- $D_{p,\text{min}} = 0.7$ μm ← note how small

1. How many G's is the particle experiencing at r_2?
2. Calculate both Q_* and the more realistic Q_{cut}.
3. Verify whether Stokes' law applies.
4. What would be the area of the sedimentation vessel that would operate at this Q_{cut}? *Hint*: recall that $A = \dfrac{Q}{v_{TSV}}$.

Example

1. Illustrate the trajectory taken by a particle reaching the cut-point within time t_{cut}
2. *In the same duration of time*, what trajectory will a smaller particle have taken?

Sigma theory for centrifuges

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

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

we obtain:

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

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

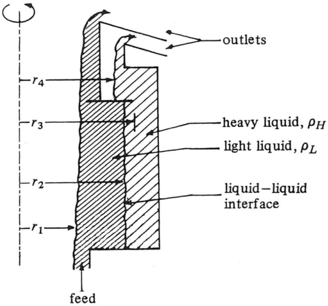
$\Sigma = f(r_1, r_2, h, \omega)$

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
- Σ has units of m^2: Σ is the equivalent surface area required for sedimentation by *gravity*
- Centrifuge A: $Q_{\text{cut},A} = v_{\text{TSV}}^{\text{grav}} \cdot \Sigma_A$
- Centrifuge B: $Q_{\text{cut},B} = v_{\text{TSV}}^{\text{grav}} \cdot \Sigma_B$
- $$\frac{Q_{\text{cut},A}}{Q_{\text{cut},B}} = \frac{\Sigma_A}{\Sigma_B}$$
- Used for scale-up **of the same feed**, i.e. the same $v_{\text{TSV}}^{\text{grav}}$
- Used for scale-up **within the same types of equipment**
- Σ equation is different for other centrifuge types
- *Question*: if I know Σ_A for a given centrifuge and for a given feed; can I calculate the performance, $Q_{\text{cut},B}$, for a different feed stream?

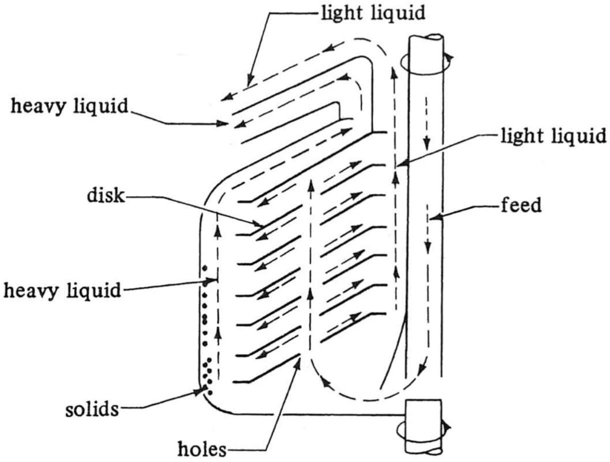
More on the tubular bowl centrifuge

- 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 is used (around 8), as it is more stable to operate
- Minimize D; very high wall stresses are developed at higher diameters
- Can be used for fluid-fluid separation



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

Disc bowl (disc stack) centrifuges



[Geankoplis, Fig 14.4-4]

Video to illustrate operation:

http://www.youtube.com/watch?v=YMbaBLpInrc

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

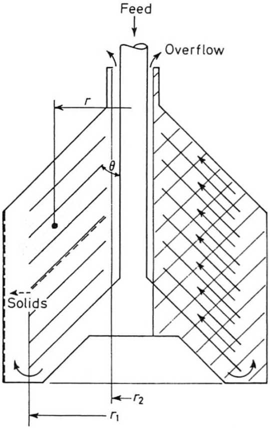
Disc bowl centrifuges

- Recall: $Q = V/t_*$ (the t_* will be different for disc bowl compared to tubular bowl)
- If we increase rate of fluid feed, we get higher throughput, Q
- Adding angled discs gives a greater surface area, hence greater volume treated, without increasing bowl diameter
- Widely used in bioseparations: no contamination (aseptic)
- Also for: fish oil, fruit juice, beverage clarification
- 3-phases separation: e.g. sand, oil, water mixtures

Disc bowl centrifuges

- Discs angled at 35 to 50°; $\sim$ 50 to 150 discs 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 discs)

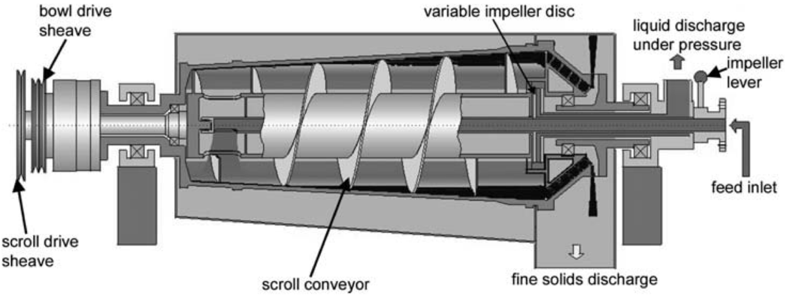
$$\Sigma = \frac{2\pi\omega^2 N(r_1^3 - r_2^3)}{3g\tan\theta}$$



- N = number of disc plates
- θ = angle of disks
- r_1 = outer cone radius
- r_2 = inner cone radius

Scroll centrifuges

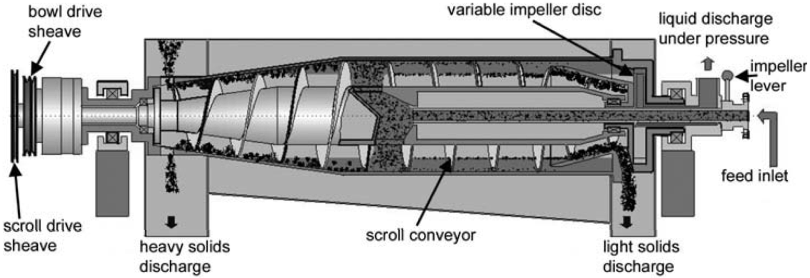
The scroll allows for continuous removal of solids:



[Perry, fig 18-159]

- Sedicanter: biotechnology, vitamin, soy, and yeast separations.

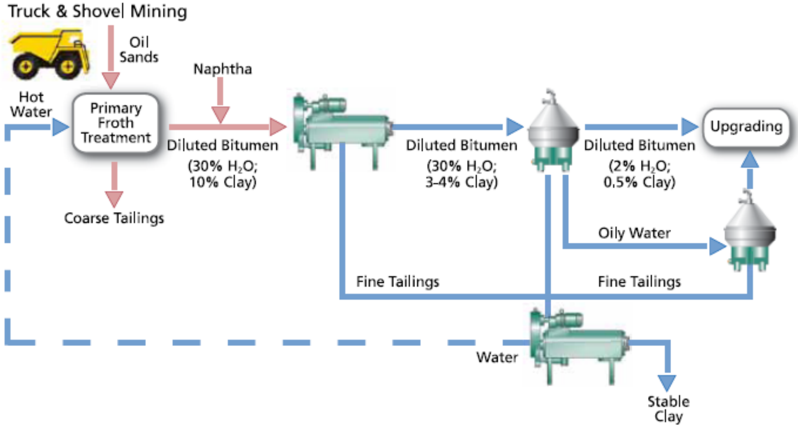
Scroll centrifuges



[Perry, fig 18-160]

- Sorticanter: used for plastics recycling
- General scroll centrifuges: used in oil-sands separations

Sequencing of centrifuges

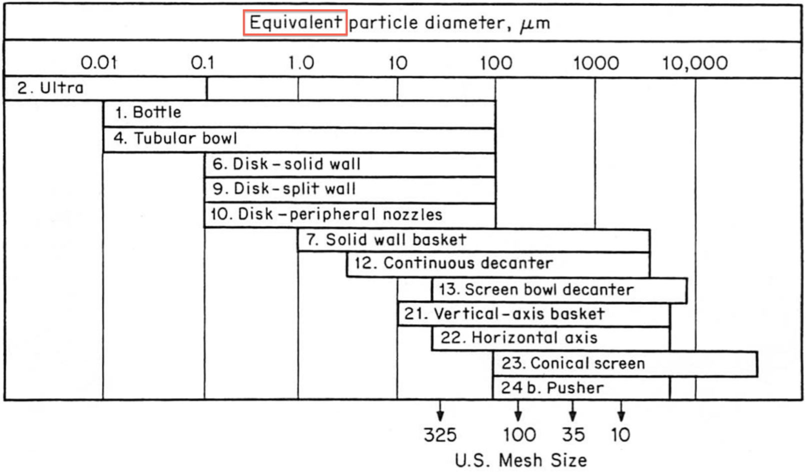


[http://www.westfalia-separator.com/products/innovations/oil-sand-bitumen-process.html]

Safety

- careful selection of materials of construction: corrosion and withstand high forces
- heat removal might be required (some units come with integrated refrigeration)
- 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

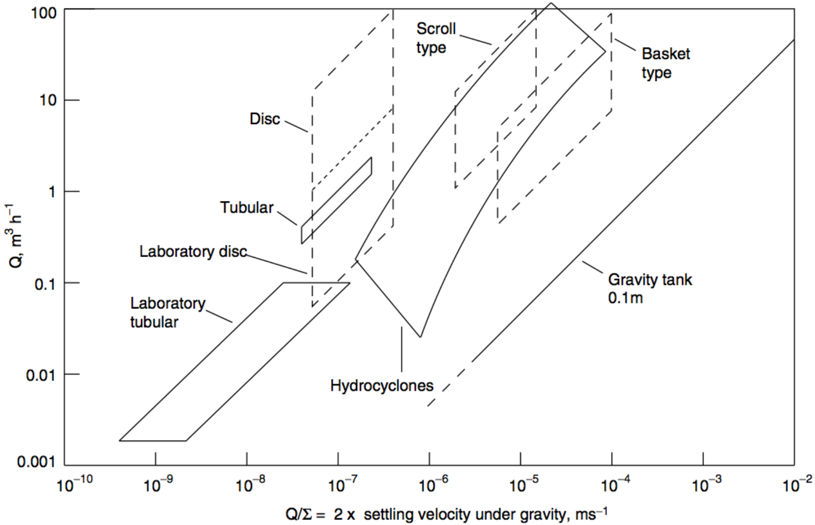
Choosing a centrifuge unit



[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 μm
- 11.5 metric tonnes of yeast are suspended in each 100 m^3 fermenter
- Aseptic operation is vital

Further practice questions

1. In a test particles of density 2800 kg.m^{-3} and of size 5 μm, equivalent spherical diameter, were separated from suspension in water fed at a volumetric throughput rate of 0.25 m^3.s^{-1}. Calculate the value of the capacity factor, Σ. [*Ans*: $\Sigma = 1.02 \times 10^4$ m^2]

2. What will be the corresponding size cut for a suspension of coal particles in oil fed at the rate of 0.04 kg.s^{-3}? The density of coal is 1300 kg.m^{-3} and the density of the oil is 850 kg.m^{-3} and its viscosity is 0.01 N.s.m^{-2}. [*Ans*: $D_{\mathrm{p,cut}} = 4\mu$m]

3. Is Stokes' law applicable? [*Ans*: Calculate the $v_{\mathrm{TSV}}^{\mathrm{cent}}$ and confirm if Re$<$ 1]

[Richardson and Harker, v2, 5th ed, p482-483]