Separation Processes

ChE 4M3

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We appreciate:

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

All of the above can be done by writing to

`kevin.dunn@mcmaster.ca`

If reporting errors/updates, please quote the current revision number: 97

Admin issues

- Group size: 2 (preferred), but groups of 3 will be allowed if you email me with a convincing explanation
- Midterm: 12 October 2012, 18:30 to 21:30; split venue
- Assignment 1 is posted on website

Mechanical separations

We will start with this topic

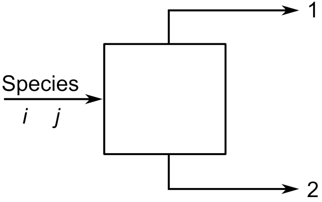
- It's easy to understand!
- Requires only a knowledge of basic physics (e.g. 1st year physics)
- It introduces a number of important principles we will re-use later
- Mechanical separations remain some of the most widely used steps in many flowsheets. Why?
  - reliable units
  - relatively inexpensive to maintain and operate
  - we can often achieve a very high *separation factor* (that's desirable!)

Separation factor

As mentioned, we will introduce a number of important principles we will re-use later.

Separation factor

$$S_{ij} = \frac{x_{i,1}/x_{j,1}}{x_{i,2}/x_{j,2}}$$



- select i and j so that $S_{ij} \geq 1$
- units of x terms in the above equation can be mass or mole fractions (or flows)

Based on this definition: we can see why solid-fluid separations often have high separation factors

Separating agents: MSA and ESA

A material, force, or energy source applied to the feed for separation

i.e. what you add to get a separation. MSA = mass separating agent and ESA = energy separating agent

- heat (ESA)
- liquid solvent (MSA)
- pressure (ESA)
- vacuum
- membrane
- filter media
- electric field
- flow
- temperature gradient
- concentration gradient
- gravitational field (natural, or artificially created)
- adsorbent
- absorbent

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Units we will consider in depth

Under the title of "Mechanical Separations" we will consider:

- free settling (sedimentation)
- screening of particles
- centrifuges
- cyclones

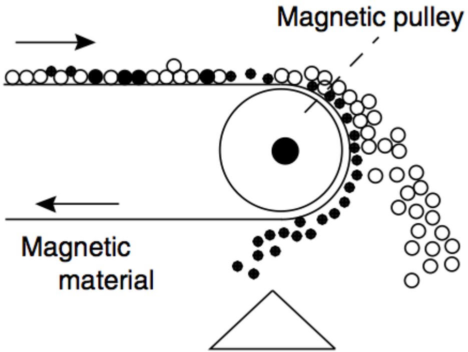
There are also others that go in this category. Deserving a quick mention are:

- magnetic separation
- electrostatic precipitation

Quick mention: Magnetic separation

- used mainly in the mineral processing industries
- high throughputs: up to 3000 kg/hour per meter of rotating drum
- e.g. remove iron from feed
- Also used in food and drug industries at multiple stages to ensure product integrity

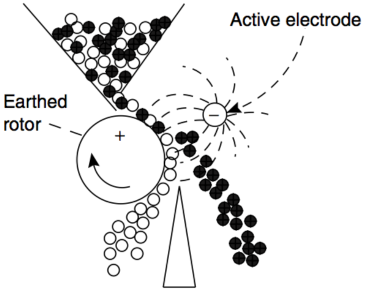
[Sinnott, 4ed, v6, Ch10]



Quick mention: Electrostatic separators

- depends on differences in conductivity of the material
- materials passes through a high-voltage field while on a rotating drum
- the drum is earthed
- some of the particles acquire a charge and adhere stronger to the drum surface
- they are carried further than the other particles, creating a split

[Sinnott, 4ed, v6, Ch10]



What is sedimentation

Class demonstration with

- concrete powder in water
- drywall compound (calcium carbonate and other particles)

DIY: add 2 to 3 tablespoons of vinegar to a glass of milk and stir

Definitions

Sedimentation

*Removal of suspended solid particles from a fluid (***liquid*** or gas) stream by gravitational settling.*

Most common to use a **liquid** rather than gas phase.

Some semantics:

Thickening: generally aims to increase the solids to higher concentration; higher throughput processes

Clarification: remove solids from a relatively dilute stream, usually aims for complete suspended-solids removal: units are deeper, and have provision for coagulation of feed.
Perry, 8ed, Ch18.5

References for this section

1. Schweitzer: Handbook of Separation Techniques
2. Svarovsky 4ed, Ch 5 and Ch 18
3. Geankoplis: 3ed and 4ed, Chapter 14
4. Perry's: 8ed, Chapter 18
5. Richardson *et al.*: Volume 2, Chapter 3 and 5

Where is it applied?

Most commonly:

- water treatment
- and mineral processing applications

But also chemical, pharmaceutical, nuclear, petrochemical processes use gravity settling to resolve emulsions or other liquid-liquid dispersions. [Svarovsky]

Topics we will cover

- factors that influence sedimentation
- designing a settler unit
- costs of building and operating a settler unit
- flocculation (coagulation)

List any factors that influence sedimentation process

- diameter of the particles
- strength of gravitational field
- relative density of particle vs fluid
- density of fluid
- viscosity of fluid
- particle concentration (hindered)
- no effect: diameter of the vessel
- no effect: mass of particle

List any factors that influence sedimentation process

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- particle concentration (hindered)
- no effect: diameter of the vessel
- no effect: mass of particle

Ideal case: momentum balance on an unhindered particle

Forces acting on a spherical particle in a fluid:

1. **Gravity**: a constant downward force $= mg = V_p \rho_p g$
2. **Buoyancy**: proportional to volume displaced $= V_p \rho_f g$
3. **Drag**: opposes the particle's motion (next slide)
4. **Particle-particle interactions** and Brownian motion: assumed zero for now

$$\begin{aligned}
V_p &= \text{particle's volume} = \frac{\pi D_p^3}{6} \text{ [m}^3\text{]} \\
\rho_p &= \text{particle density [kg.m}^{-3}\text{]} \\
\rho_f &= \text{density of fluid [kg.m}^{-3}\text{]} \\
\mu_f &= \text{fluid's viscosity [Pa.s]} \\
g &= \text{gravitational constant} = 9.81 \text{ [m.s}^{-2}\text{]} \\
D_p &= \text{particle's diameter [m]}
\end{aligned}$$

Drag force

$$F_{\text{drag}} = C_D A_p \frac{\rho_f v^2}{2}$$

where

$$\begin{aligned} v &= \text{relative velocity between the particle and the fluid } [\text{m.s}^{-1}] \\ A_p &= \text{projected area of particle in direction of travel } [\text{m}^2] \\ C_D &= \text{drag coefficient (it's assumed constant!) } [-] \end{aligned}$$

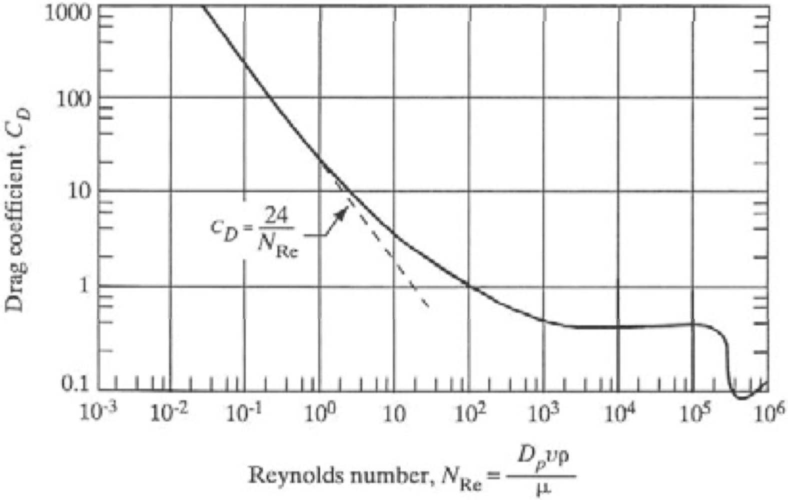
Estimating the drag coefficient

It's a function of Reynolds number $= \text{Re} = \dfrac{D_p v \rho_f}{\mu_f}$

[Richardson and Barker, p 150-153]

1. If $\text{Re} < 1$
$$C_D = \frac{24}{\text{Re}}$$
2. If $1 < \text{Re} < 1000$
$$C_D = \frac{24}{\text{Re}}\left(1 + 0.15\text{Re}^{0.687}\right)$$
3. If $1000 < \text{Re} < 2 \times 10^5$
$$C_D = 0.44$$
4. If $\text{Re} > 2 \times 10^5$
$$C_D = 0.10$$

Drag coefficient as a function of Re



Geankoplis, 3rd p818, 4th p921

Momentum balance (Newton's second law)

$$m\frac{dv}{dt} = F_{\text{gravity}} - F_{\text{buoyancy}} - F_{\text{drag}} = 0 \qquad \text{at steady state}$$

$$0 = V_p\rho_p g - V_p\rho_f g - C_D A_p \frac{\rho_f v^2}{2}$$

Substitute $V_p = \dfrac{\pi D_p^3}{6}$ and $A_p = \dfrac{\pi D_p^2}{4}$ for spherical particles and solve for v:

Terminal velocity of an unhindered particle

$$v = \sqrt{\frac{4\left(\rho_p - \rho_f\right) g D_p}{3 C_D \rho_f}}$$

Stokes' law

Simplification of the above equation when Re < 1:

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

Confirm it for yourself: *hint*: use the solution for a quadratic equation $ax^2 + bx + c = 0$

Solving the general equation for v

$v = fn(C_D)$, but $C_D = fn(\mathrm{Re}) = fn(v)$

1. Assume $\mathrm{Re} < 1$ (Stokes' region)
2. Solve for v
3. Calculate Reynolds number, Re
4. Was Reynolds number region assumption true? If so: stop.
5. If not, use new Re and recalculate C_D
6. Repeat from step 2 to 5 until convergence

Alternative approach shown in Geankoplis.

Why is the terminal velocity so important?

Design criterion

Terminal velocity of the slowest particle is our limiting design criterion

Hindered settling

Particles will not settle as perfect spheres at their terminal velocity under a variety of conditions:

- if they are hindered by other particles
- they are non-spherical
- concentrated feeds: particles form clusters that tend to settle faster
- concentrated feeds: modify the apparent density and viscosity of the fluid
- upward velocity of displaced fluids
- small particles are dragged in the wake of larger particles
- ionized conditions can cause particle coagulation → larger diameters → faster settling

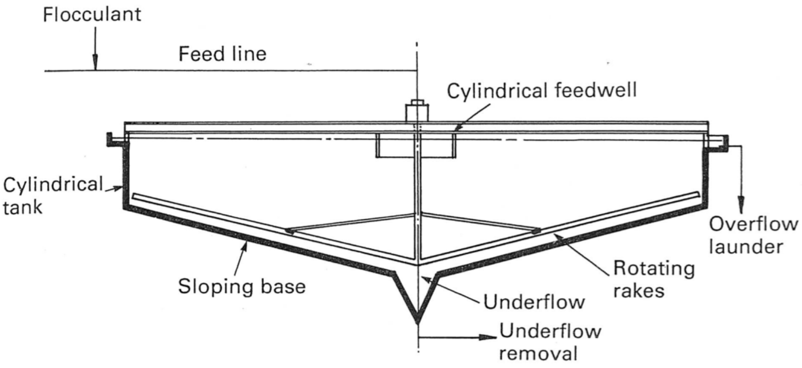
Video: http://www.youtube.com/watch?v=h8n3Nt4tPXU
How to deal with this issue?

Video of sedimentation experiments

http://www.youtube.com/watch?v=E9rHSLUr3PU

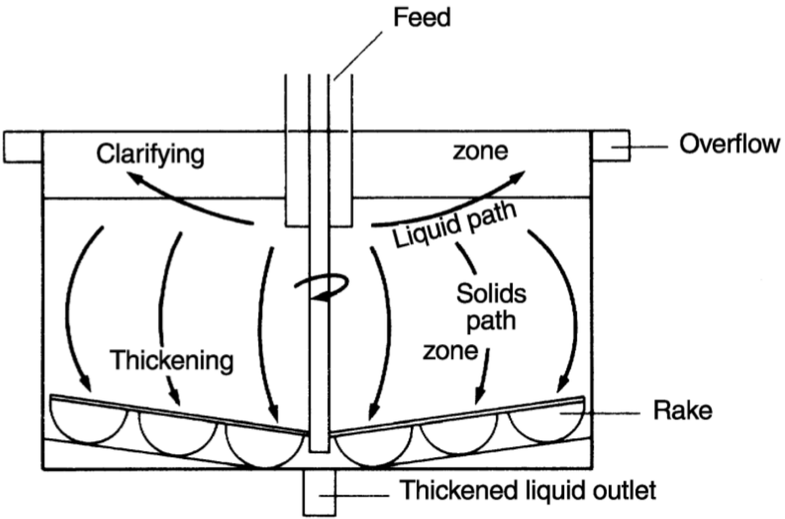
Settler terminology

A standard gravitational thickener:



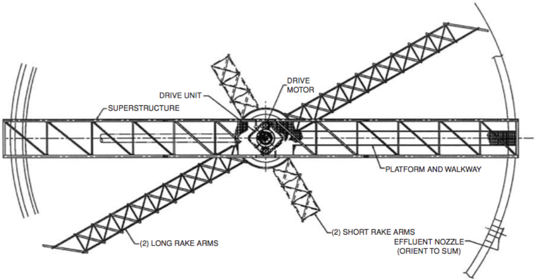
[Svarovsky, 3ed, p141]

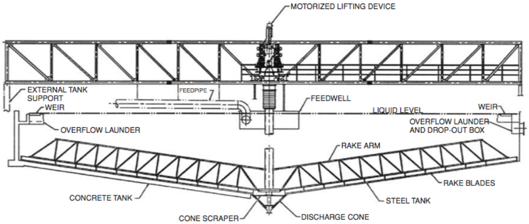
Gravitational thickener terminology



[Richardson and Harker, 5ed, v2, p256]

Gravitational thickener top and side view





[Perry's, 8ed, 18-71]

Unhindered settling

- Takes place when settling occurs at a constant rate, independent of other particles.
- Use the equations derived in last class to estimate settling velocity $= v$.
- Draw an imaginary horizontal layer through the settler and observe the mass of solids passing across it per unit time, per unit area = mass flux.
- The flux of solids is $\psi = C_0 v$, with units of $\dfrac{\text{kg solids}}{\text{m}^3\ \text{feed}} \cdot \dfrac{\text{meters}}{\text{second}}$
- $\psi = C_0 v \quad \dfrac{\text{kg solids}}{\text{second}} \cdot \dfrac{1}{\text{meters}^2}$
- ψ = mass feed rate per unit area = loading rate
- $\dfrac{1}{\psi}$ = unit area required per given amount of mass feed rate

Note: assuming no solids leave the overflow

Preliminary settler area estimate

The area required under these ideal conditions:

$$A = \frac{QC_0}{\psi} = \frac{QC_0}{C_0 v} = \frac{Q}{v}$$

where

- Q = volumetric feed rate [m^3 of feed per unit time]
- C_0 = concentration of solids in feed [kg of solids per m^3 feed]
- v = settling velocity [meters per unit time]

Example

A sample of material was settled in a graduated lab cylinder 300mm tall. The interface dropped from 500mL to 215mL on the graduations during a 4 minute period.

1. Give a preliminary estimate of the clarifier diameter required to treat a waste stream of 2100 L per minute. Over-design by a factor of 2, based on the settling rate, and account for about 7 m^2 of entry area used to eliminate turbulence in the entering stream.
2. If the feed concentration is ~~200~~ 3.5 kg per m^3 feed, what is the loading rate? Is it within the typical thickener range of 50 to 120 kg per day per square meter? [Perry, 8ed, p22-79]

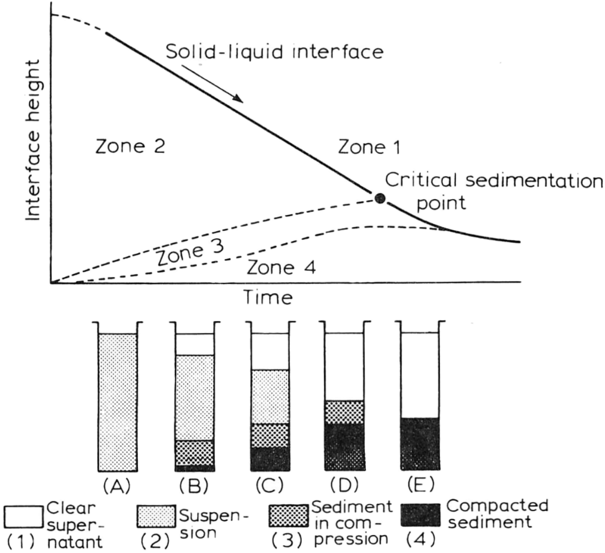
Answers:

1. Settling rate = 171 mm per 4 minutes = 42.8 mm/min.

$$\text{Area} = \frac{2.1\ \text{m}^3.\text{min}^{-1}}{(0.5)\left(42.8 \times 10^{-3}\text{m}.\text{min}^{-1}\right)} = 98 + 7\text{m}^2$$

2. $\psi = C_0 v = 3.5 \dfrac{\text{kg}}{\text{m}^3} \cdot 0.022 \dfrac{\text{m}}{\text{min}} \cdot \dfrac{60 \times 24\text{min}}{\text{day}} = 106 \dfrac{\text{kg}}{\text{day.m}^2}$

Setting zones during sedimentation



[Svarovsky, 3rd ed, p 135]

Hindered settling

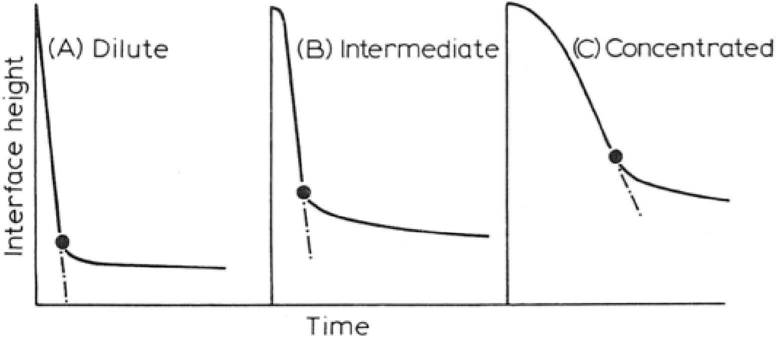
For a high concentration of particles we have hindered settling:

- form clusters that settle faster
- however, there is upward flow from displaced fluids
- apparent density and viscosity of the fluid phase is increased
- drag force increased due to other particles

Approaches to dealing with this:

1. Modify the density, viscosity and other terms in the momentum balance: use correction factors
2. Resort to lab tests on samples that closely match the actual feed material
    - use lab results to design the settler

The effect of particle concentration



More concentrated solutions take longer to settle; sometimes see clearer supernatants with concentrated solutions: small particles are pulled down in wake of larger particles.

How can we accelerate settling?

- modify the particle shape: spherical vs needle shape (usually not possible)
- modify the fluid viscosity and density
  - not practical in most cases
  - e.g. used to separate diamonds in a process called "dense medium separation"
- raking or stirring: creates free channels for particles to settle in
- flocculation: to increase the particle's size by coagulating particles

Flocculation

MIT video on water cleaning:
http://www.youtube.com/watch?v=5uuQ77vAV_U

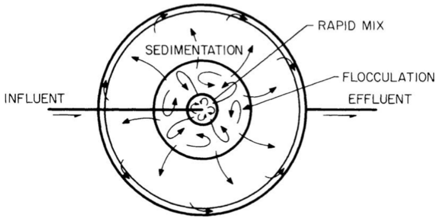
Please take notes from the video

Flocculation

Small particles (around $< 40\mu m$) and some biologically active particles will take unreasonably long times to settle, if at all. Flocculated particles cluster together and settle at higher rates

- impossible to predict shape and hence settling rate
- used in clarifiers, where clear supernatant is desired

Flocculation is commonly "included" with the sedimentation step:

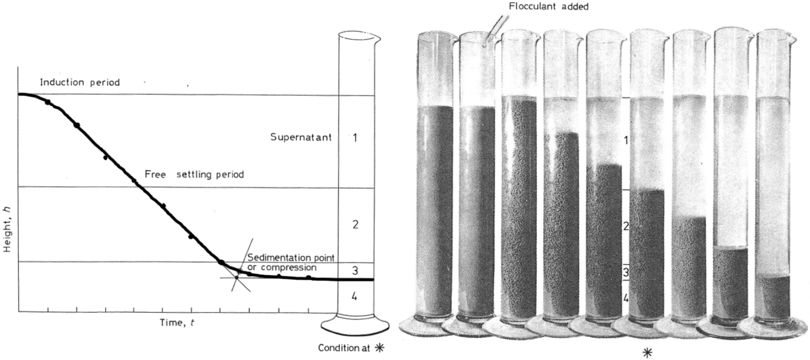


[Perry, 8ed, Ch22]

- important to not disrupt the flocs after contacting with flocculant: 30 seconds to 2 minutes contact time

Sludge interface experiments

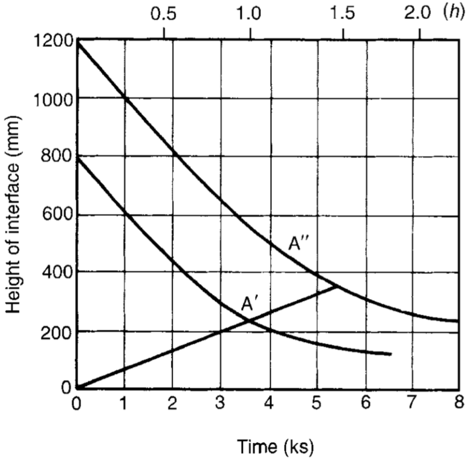
Since flocculant and concentration effects cannot be derived from theory, resort to lab settling tests.



Sludge interface experiments

Interesting point

For a given feed, if you have the settling curve for one height, you have it for other heights also. Ratio 0A' : 0A" is constant everywhere.



Sludge interface experiments

- initial constant rate of settling is observed
- a critical point is reached: point of inflection
- slow compression of the solids after this point

At least 2 procedures in the literature to design settlers from settling curves:

- Talmage and Fitch: tends to overdesign the area
- Coe and Clevenger: underdesign of the thickener area

[Svarovsky, 4ed, p 180]

In practice: we will rely on outside consultants and civil engineers, most likely, to size and design the unit. Else see the references at end for more details.

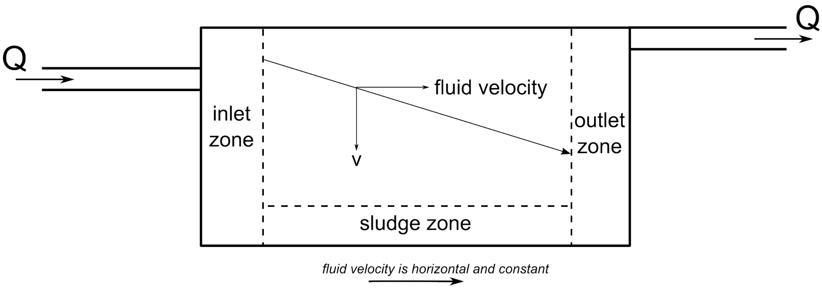
Settler design: shape, length, width

1. What width and depth should the settler be?
2. How long should the particles be in the settler? Does residence time matter?

Perry's, section 22.5.6:

- sedimentation tank can be rectangular or circular
  - rectangular: effluent weirs at the end
  - circular: around the periphery
- main concern: uniform flow in the tank (no short-circuits)
- removal efficiency = f(hydraulic flow pattern in tank)
  - incoming flow must be dissipated before solids can settle
  - evenly distributed; minimal disruption to existing fluid
  - overflow and underflow draw collected without creating hydraulic currents
  - solids are removed by scraping, and hydraulic flow

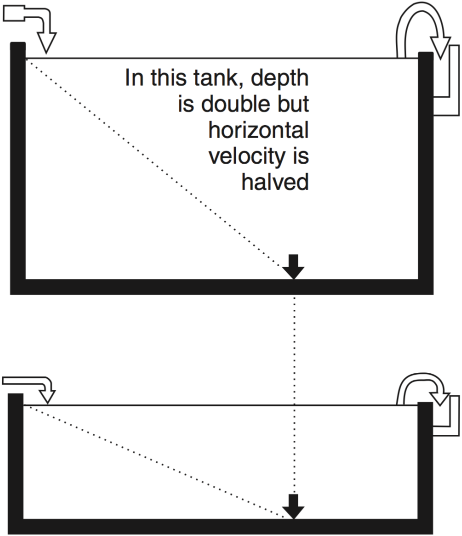
Concept: the ideal rectangular settling basin



- Inlet zone: feed is assumed to be uniformly distributed across the tank's cross-section (if viewed from the top)
- Settling zone: where particles move downwards towards the sludge area; particles also move horizontally due to fluid flow
- Outlet zone: the supernatant/clarified liquid is collected along the basin's cross section and removed in the *overflow*
- Sludge zone: where the solids collect and are removed in the tank's *underflow*

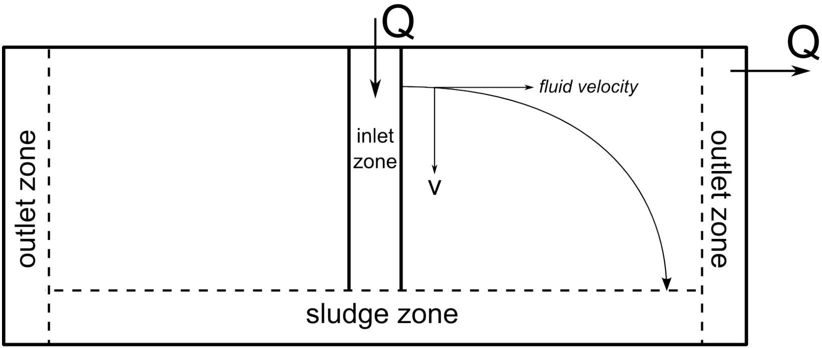
Ensure horizontal fluid velocity (i.e. residence time) is slow enough that particles at their terminal velocity, v, will reach the sludge zone and settle out.

The ideal rectangular settling basin



Changing depth has no effect in a rectangular basin [Svarovsky, 4ed, p170]

Concept: the ideal circular settling basin



fluid velocity is not constant; has a radial profile

- Same zones as before
- Fluid velocity is a function of radial distance
- As before, ensure residence time is long enough for particles to reach the sludge zone

Settler design rules of thumb: size

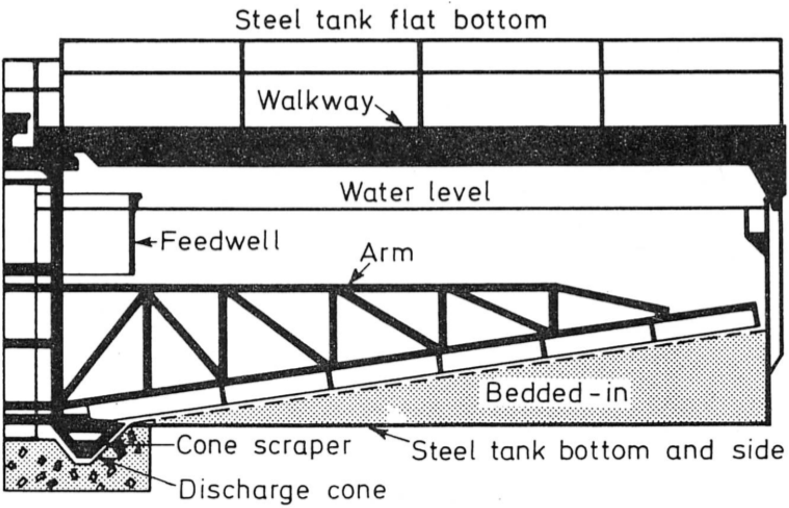
For wastewater treatment the **main design criterion**: solids percentage in underflow

- A volume and mass balance on solids and liquids is then used to find the liquid overflow rate
- surface overflow rate (SOR) $\sim 40\text{m}^3$ per day per m^2 for primary units
- secondary units as low as 12 up to 30m^3 per day per m^2
- minimum depth of sedimentation tanks is around 3.0 m
- circular sedimentation: minimum diameter of 6.0 m
- length to width ratio of 5:1

Settler design rules of thumb: residence time

- gravity sedimentation tanks normally provide for 2 hour retention of solids, based on average flow
- longer times for light solids, or in winter times
- organic solids generally will not compact to more than 5 to 10%
- inorganic solids will compact up to 20 or 30%
- why important: we have to design sludge pumps to remove the solids: high concentration solids require diaphragm pumps

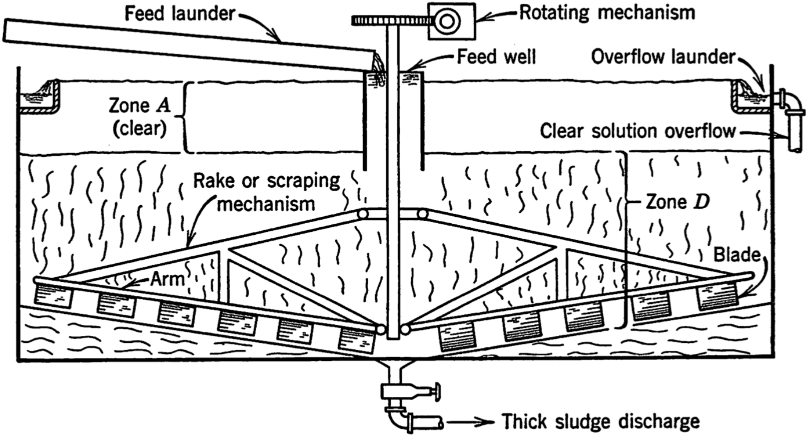
Some thickener designs



[Svarovsky, 3ed, p172]

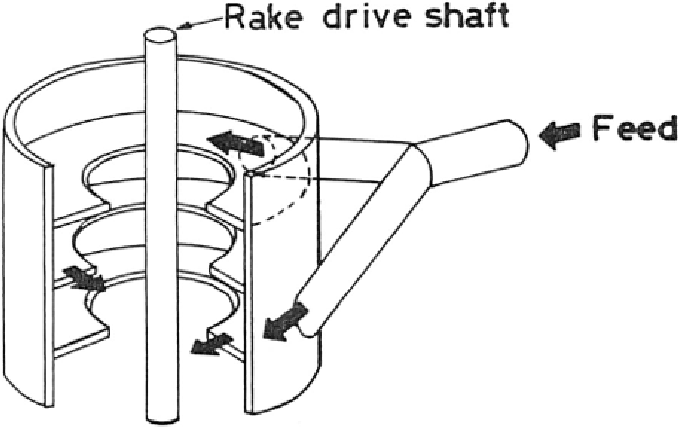
Can replace the steel with a sloped concrete slab.

Another picture: use in 2013



Feed area: feedwell

- aim to minimize turbulence from entry velocity
- avoid disruption to existing settling
- avoid breaking up existing flocs
- must not get clogged



Capital costs considerations

Svarovsky 3rd, p179: cost $= ax^b$

- $x =$ tank diameter between 10 and 225 ft
- $a = 147$ and $b = 1.38$ for thickeners

Perry, 8ed, section 18.6

- Installation costs will be at least 3 to 4 times the actual equipment costs.
- Equipment items must include:
  - rakes, drivehead and motors
  - walkways and bridge (center pier) and railings
  - pumps, piping, instrumentation and lift mechanisms
  - overflow launder and feed

  Installation is affected by:
  - site surveying
  - site preparation and excavation
  - reinforcing bar placement
  - backfill

Operating costs

These are mostly insignificant

- e.g. 60 m (200 ft) diameter thickener, torque rating = 1.0 MN.m: requires $\sim$ 12 kW
- due to slow rotating speed: peripheral speed is about 9 m/min
- implies low maintenance costs
- little attention from operators after start-up
- chemicals for flocculation (if required), frequently dwarfs all other operating costs [Perry, 8ed, Ch18.6]

Further self-study

- Designs with peripheral inlets (submerged-orifice flow control) and either center-weir outlets or peripheral-weir outlets adjacent to the peripheral-inlet channel.
- Deep cone thickener
- Lamella (inclined plate or tubes): often for gas-solid applications

Further references to explore

- Talmage and Fitch, 1955, "Determining Thickener Unit Areas", *Ind. Eng. Chem.*,**47**, 38-41, DOI:10.1021/ie50541a022
- Fitch, 1965, "Current theory and thickener design", *Ind. Eng. Chem.*, **57**, p 18-28, DOI:10.1021/ie50682a006
- Svarovsky, "Solid Liquid Separation", 3rd or 4th edition. Particularly thorough regarding the settler's mechanical accessories: pumps, scrapers, *etc.*

Practice questions

1. Calculate the minimum area and diameter of a circular thickener to treat 720 m^3 per hour of slurry containing 20μm particles of silica, whose density is about 2600 $kg.m^{-3}$. The particles are suspended in water at a concentration of 650 $kg.m^{-3}$. The slurry cannot be tested in a lab. Use an over-design factor of 1.5 on the settling velocity.
2. If it is desired to have an underflow of 1560 kg solids per m^3 underflow; what is the underflow volumetric flow rate if total separation of solids occurs?
3. Calculate the separation factor.