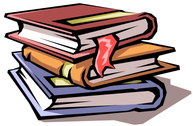
Filtration, sedimentation, centrifugation,

Institute of Pharmaceutical Technology and Biopharmacy



BASIC OPERATIONS

FILTRATION

Filtration

Filtration is an **operation** (separation process), in which

a **heterogeneous mixture can be separated** to its different forms of components

solid, liquid, gas).

Filtration is applied in case of:

- crystallization
- fiber examination of injections
- cleaning of air

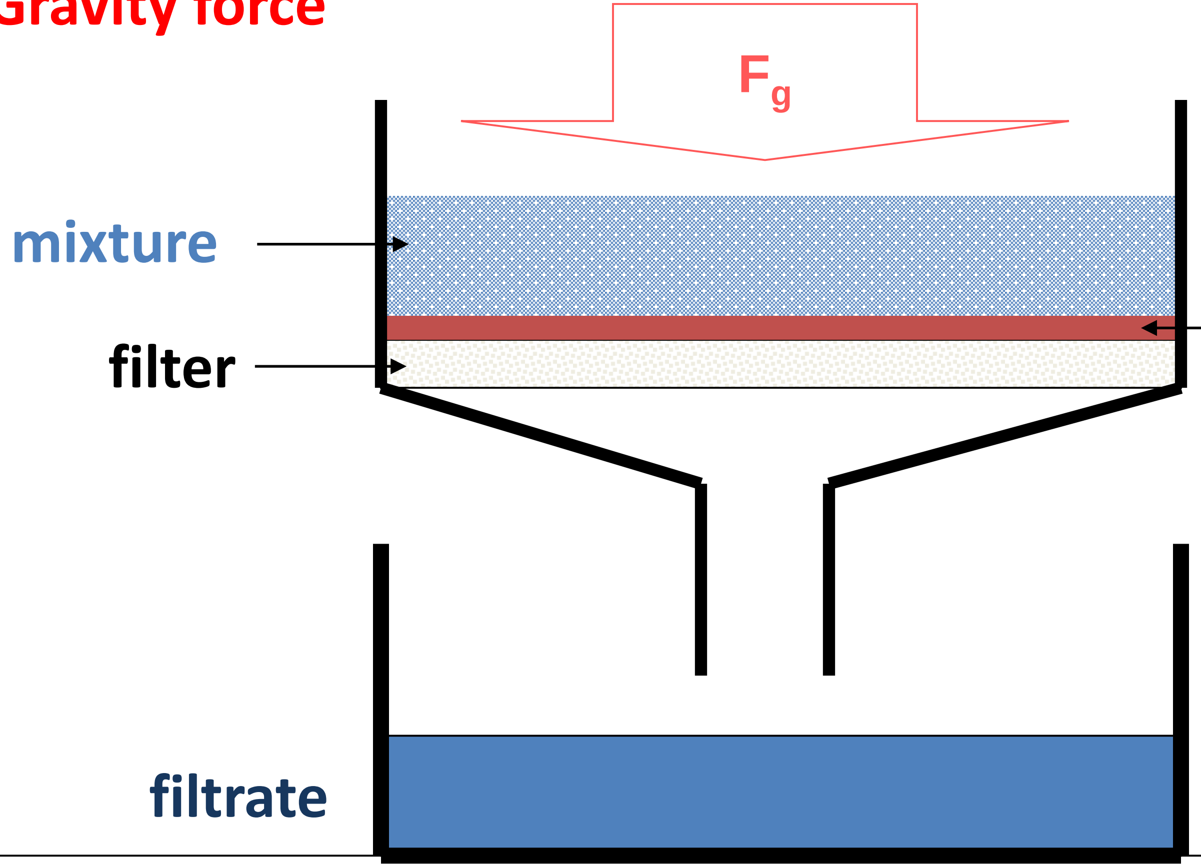
Filtration

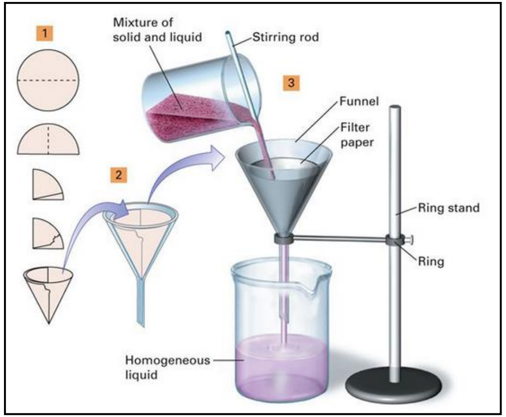
The **driving forces** of filtration:

- gravitation,
- pressure or suction,
- centrifugal force.

Filtration

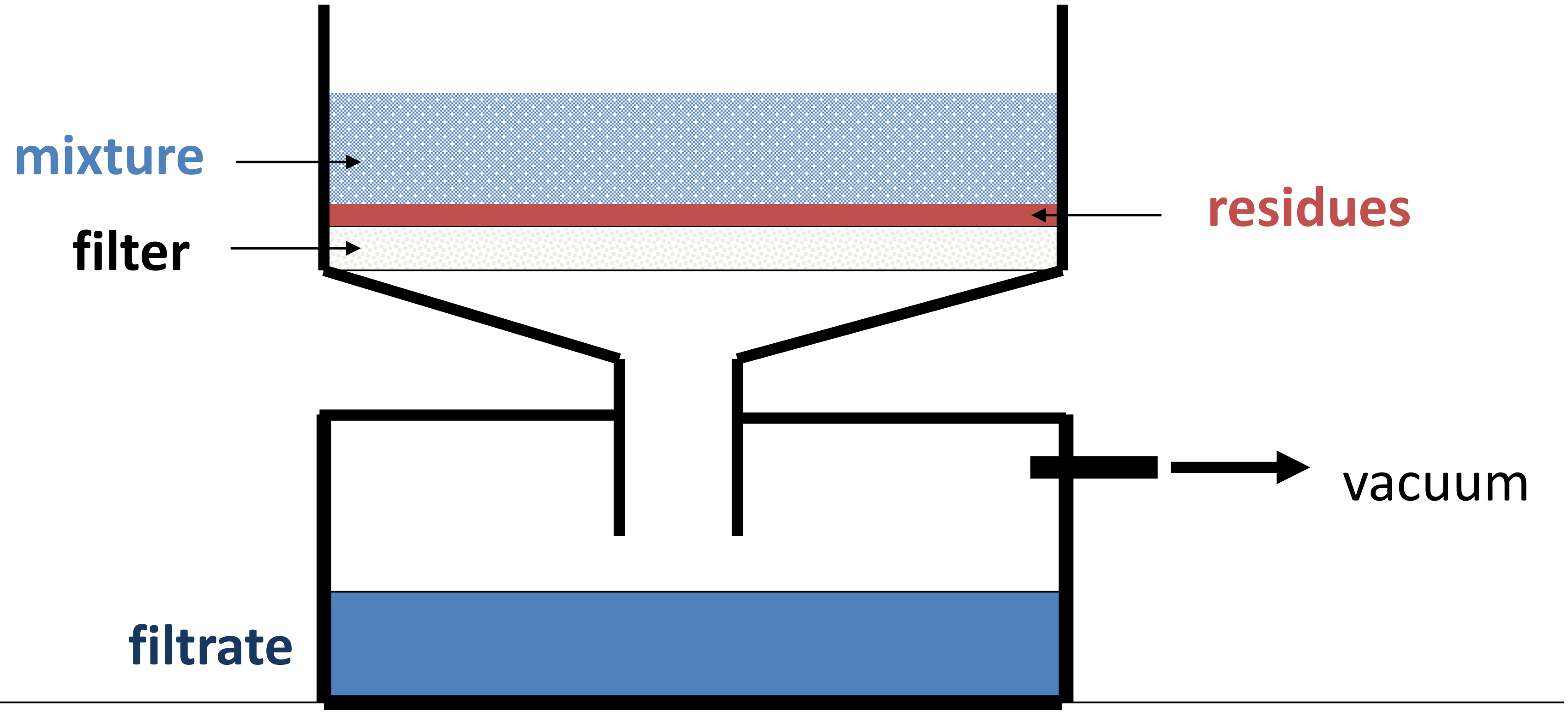
Gravity force





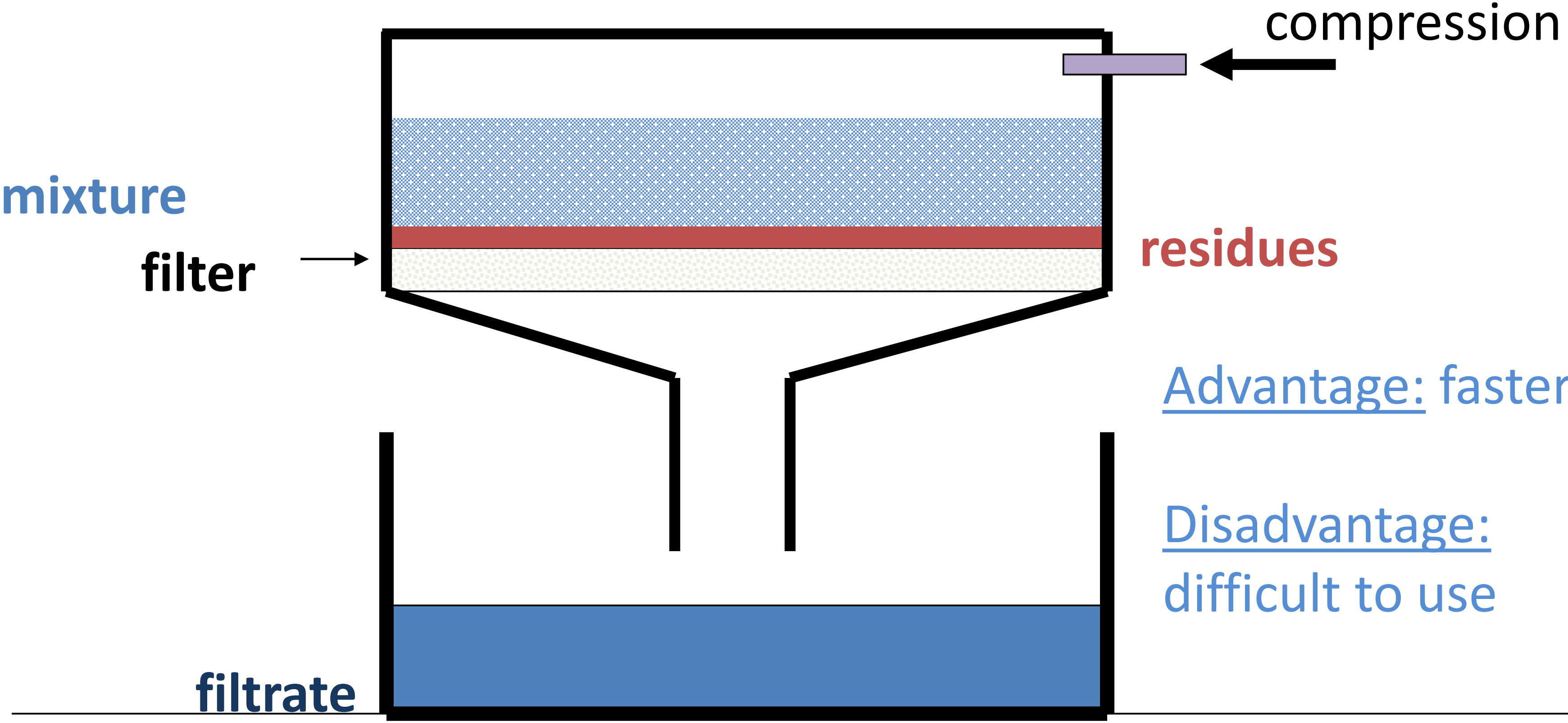
Filtration

Vacuum (suction)



Filtration

Pressure



Advantage: faster

Disadvantage:
difficult to use

Filtration rate

The filtration rate depends on:

- **thickness of the bed**
- **permeability**

Filtration rate

The **residue** may be

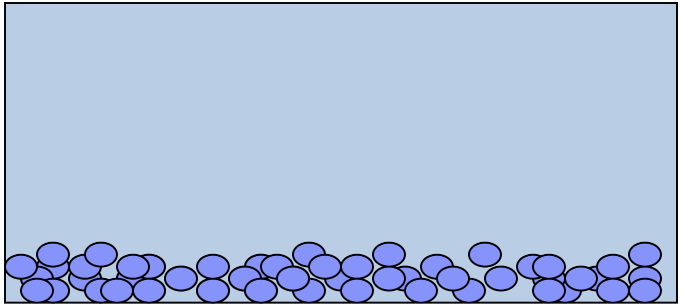
- **Incompressible**
  - the size of pores and canals is constant,

- **Compressible**
  - the size of pores and canals is decreasing during the filtration.

Filtration rate

Structure of the residue (1):

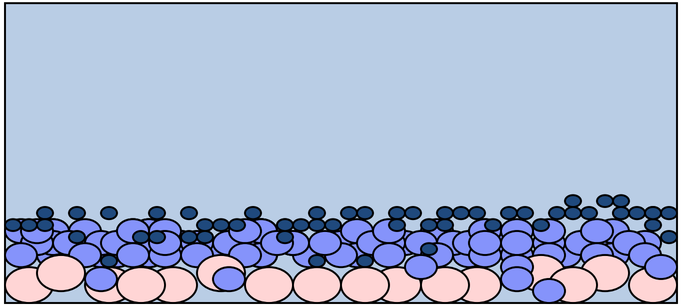
- homodisperse,
- the filtration rate is constant,
- no compaction.



Filtration rate

Structure of residue (2):

- heterodisperse
- the filtration rate is decreasing during the process,
- compaction



Filtration rate

Darcy equation

$$\frac{dV}{dt} = \frac{BA\Delta p}{\eta L}$$

V	=	*volume of the filtrated mixture*
t	=	*time*
B	=	*permeability*
A	=	*area of the filter*
Δp	=	*difference of the pressure*
η	=	*viscosity*
L	=	*thickness of the filter*

Filtration rate

Kozeny-Carman equation

(fibrous filter media)

$$B = \frac{d_f^{\,2} . \varepsilon^3}{16(1-\varepsilon)^2 k}$$

B	=	*permeability*
d_f	=	*filter diameter*
ε	=	*porosity*
k	=	*Kozeny-Carman-constant*

Filtration rate

Meyer-Smith equation

(granular filter medium)

$$\boxed{B = \frac{d_{sp}^{\,2} . \varepsilon^{4,1}}{150(1-\varepsilon)^2}}$$

B = *permeability*

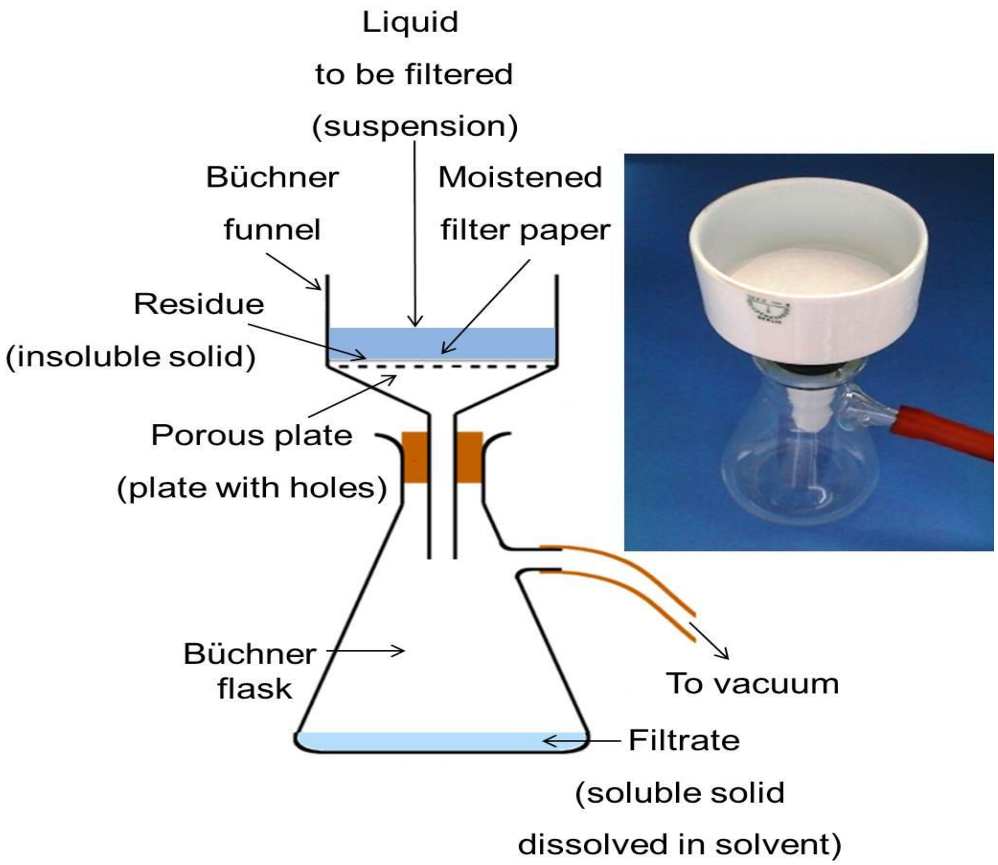
d_{sp} = *mean granule diameter*

ε = *porosity*

Equipments

Vacuum

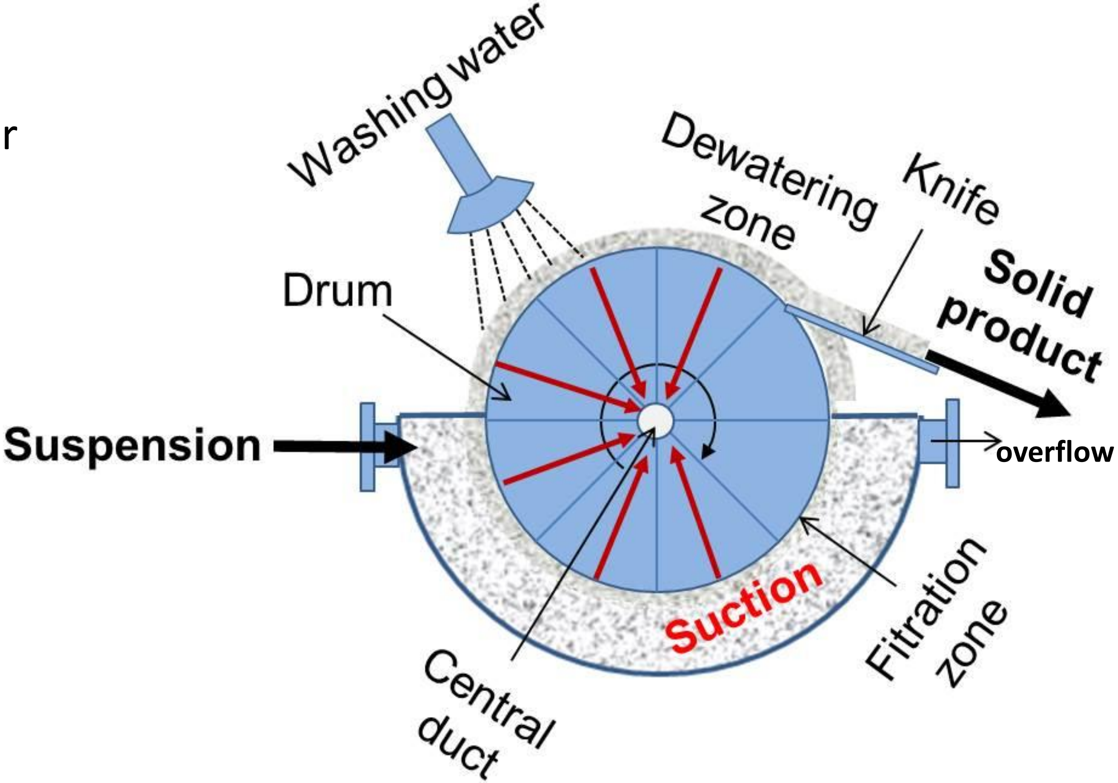
laboratory
suction filter



Equipments

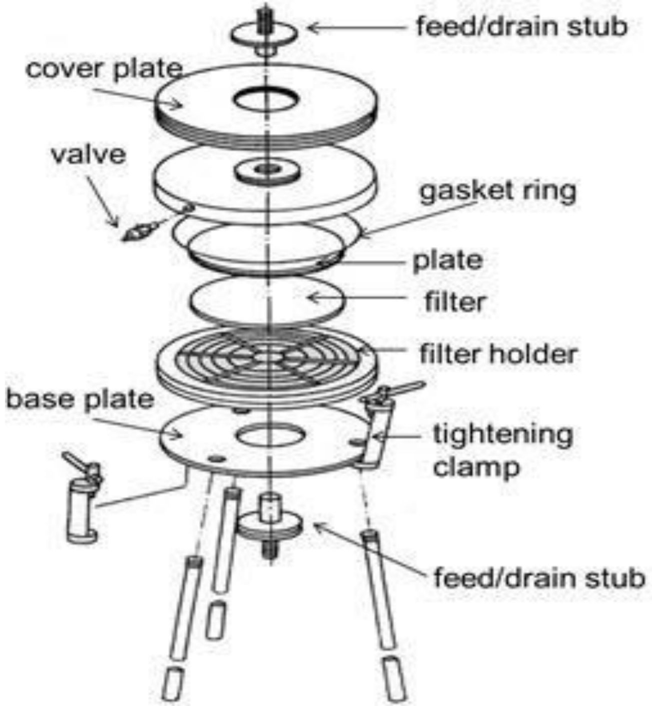
Vacuum

Vacuum
rotary-drum filter



Equipments

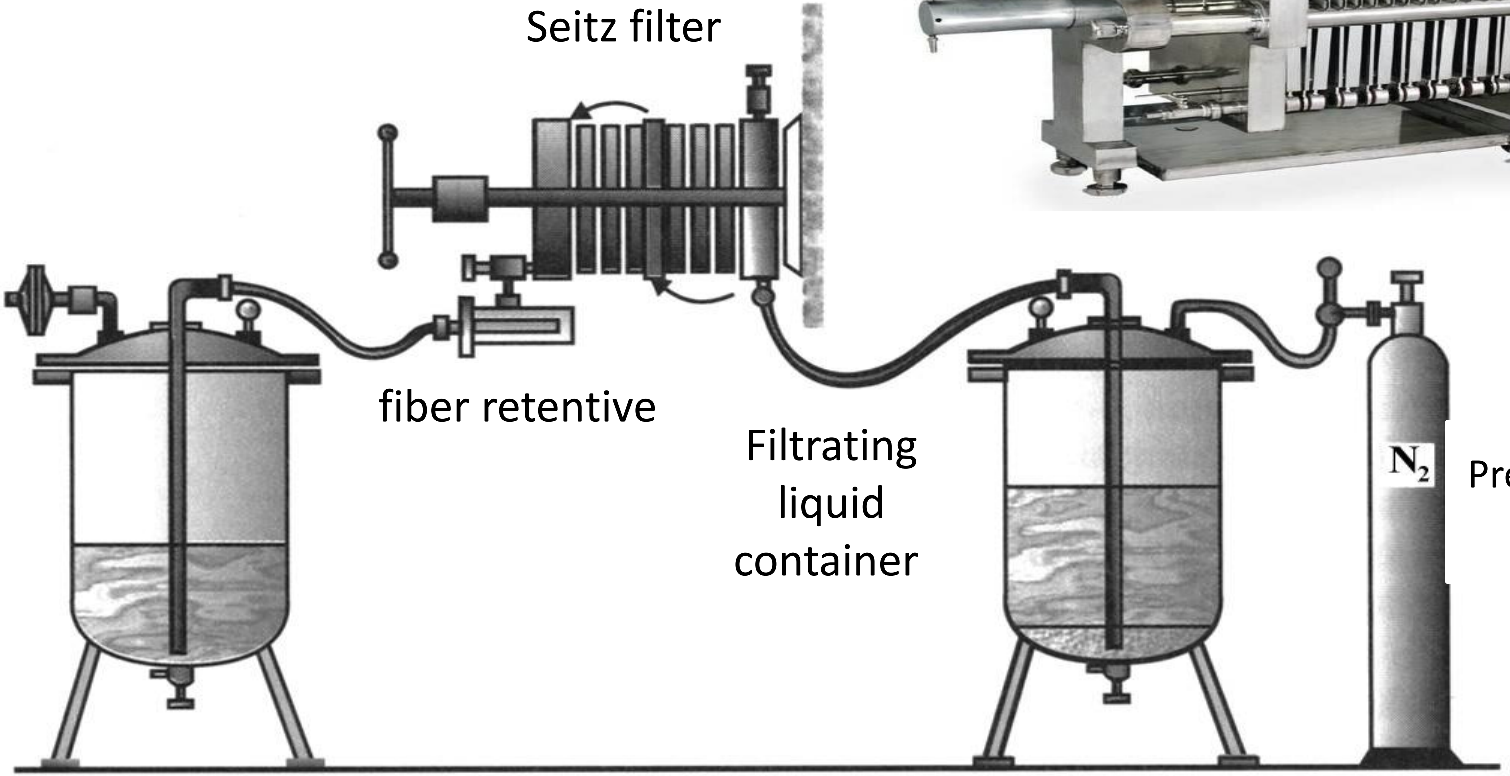
Pressure



laboratory

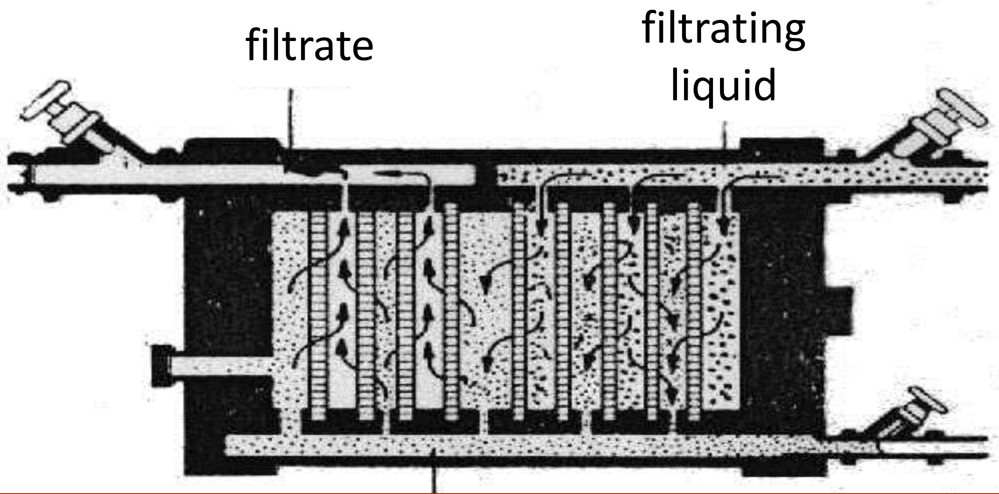
Equipments

Frame and plate filter

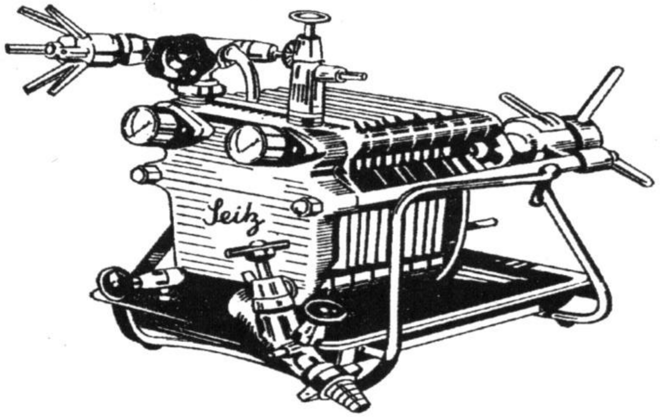


Equipments

Double filter press

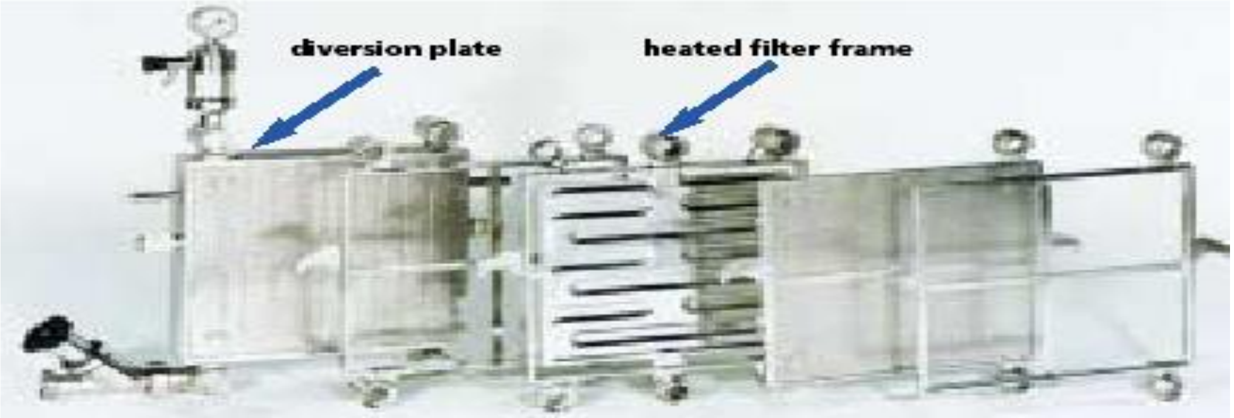


frame filter



Equipments

Frame filter

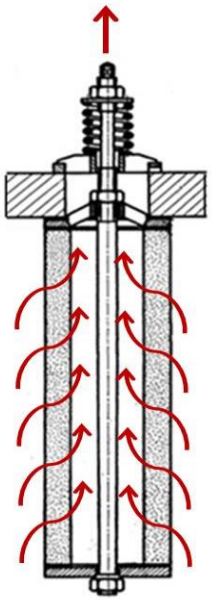


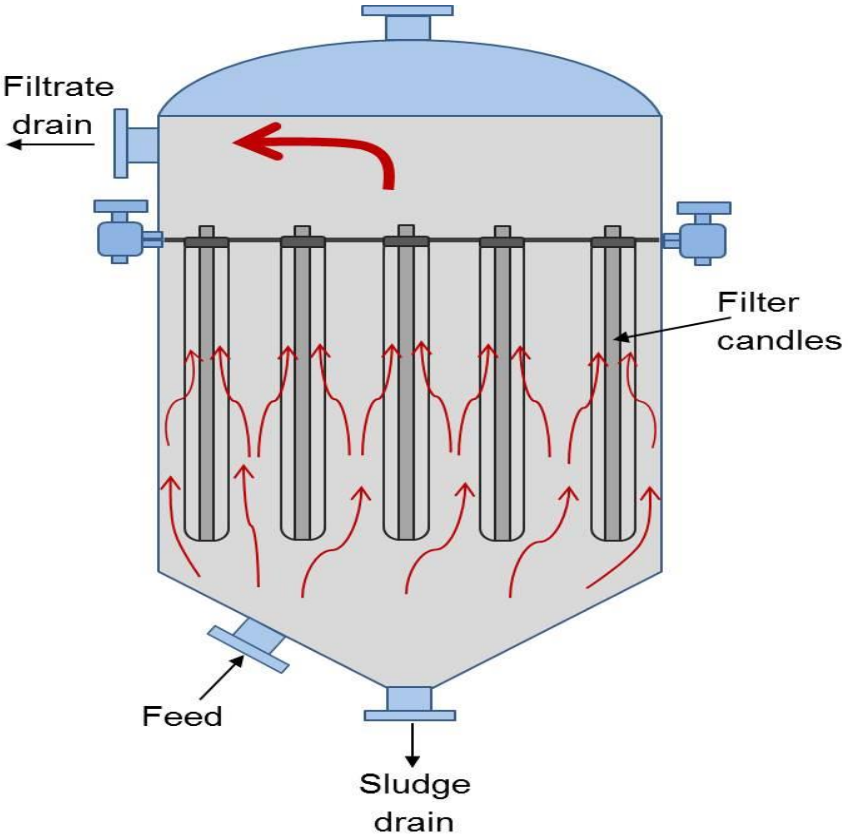
laboratory

industrial

Equipments

Candle filter

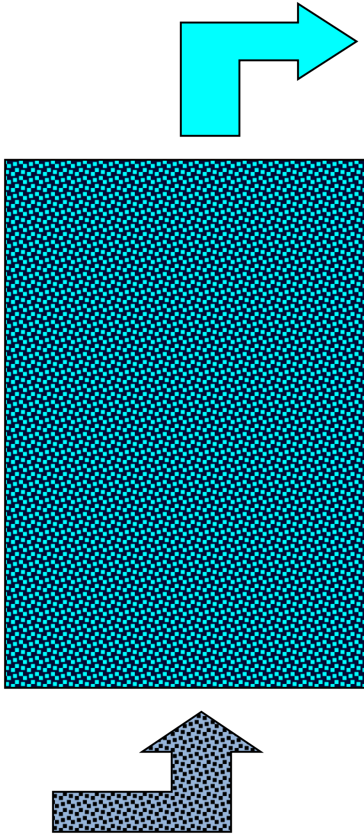




Equipments

Column filter

Filtrate



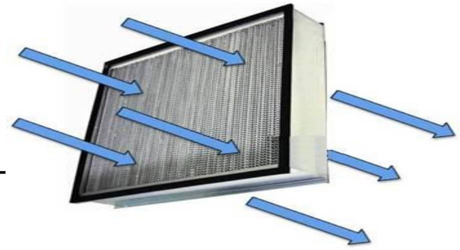
Filtered mixture

capillar effect

Filled with granules or adsorptive substances (gravel, pellets)

Seitz-filters

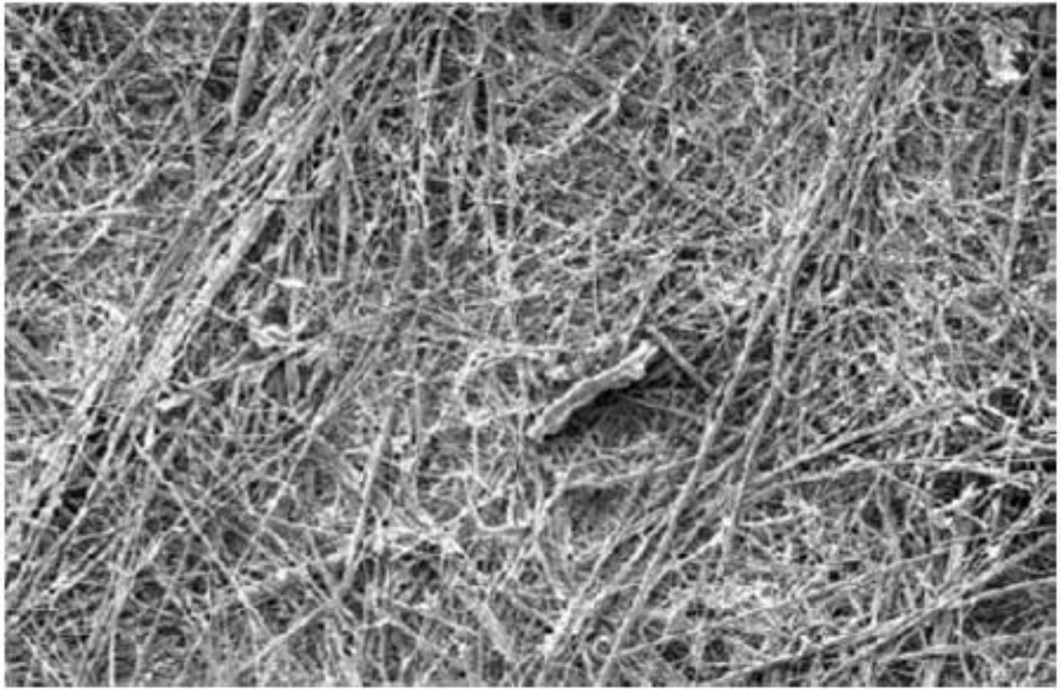
Cellulose filters

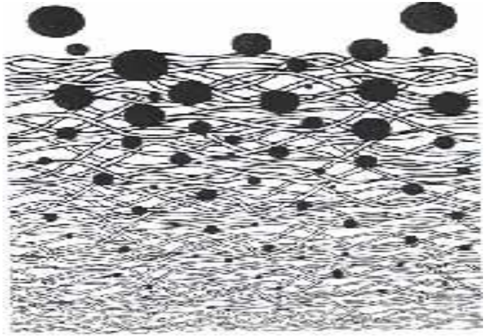


Types of Seitz-filter	Pore size (µm)	Filter capacity (ml/h)		Application
		19,62 kPa	49,05 kPa	
EK	1,4 – 1,8	200	500	**Germ-free drinks (galenical preparations, syrups, extracts)**
DKS	1,2 – 1,4	130	325	**Germ-, and pyrogen-free water, injections (aqueous), low molecular weight solution**
EKS-1	1,0 – 1,2	100	250	**Injections (aqueous), kolloidal solutions or higher molecular weight solutions (proteins), blood replacement**
EKS-11	0,8 – 1,0	75	190	**See: EKS-1, especially by blood and serums**

Depth filters

Depth (fibered) filters





Filtration trought the depth filter

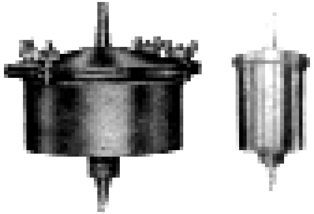
Filters

Rigid, porous filters

- *Porous ceramic* (Chamberlain filters, ~candles - kaoline
  Berkefeld filters - diatomaceous earth)
  clarifying and sterilizing filtration

- *Glass filters* (Pyrex, Schott - sintered glass filter)

  clarifying and fiber-exception and sterilizing filtration



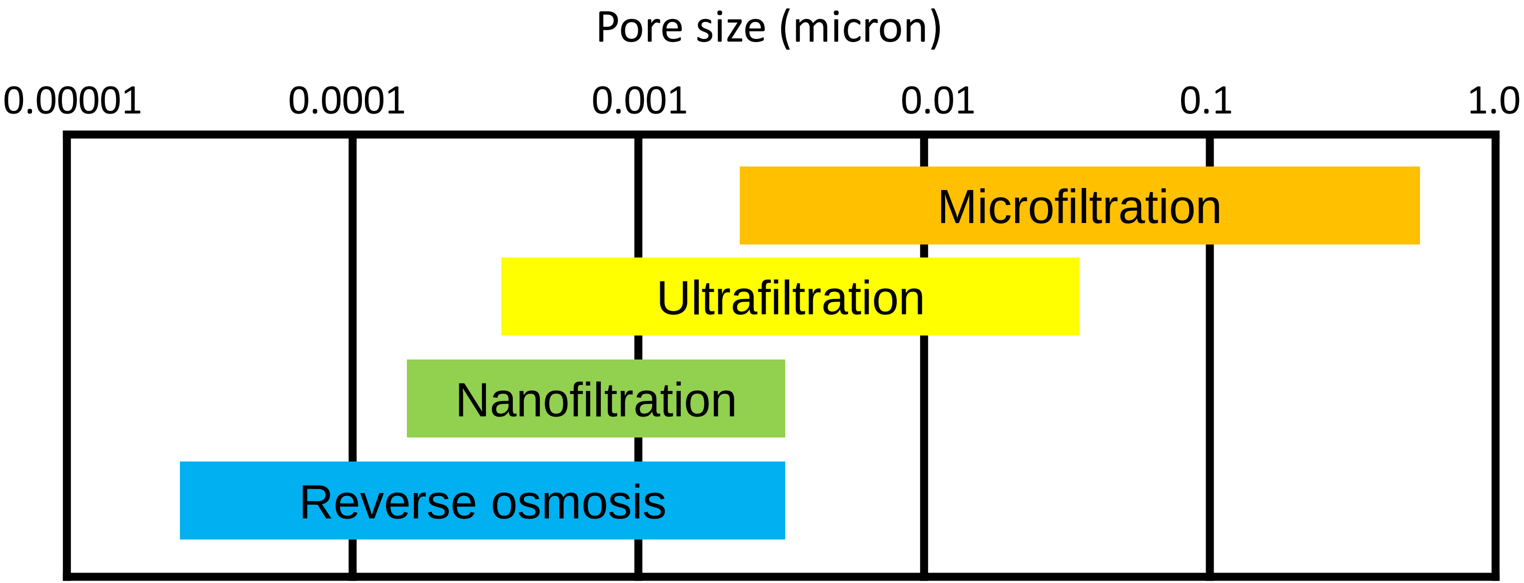
Properties of Chamberland filters

Types	Pore size (µm)	Application
Chamberland L 1	4,7 – 8,9	Clarify
Chamberland L 2	2,2 – 4,7	Clarify
Chamberland L 3	2,0 – 2,2	Clarify
Chamberland L 5	1,0 – 2,0	Sterilization
Chamberland L 7	1,0	Sterilization
Chamberland L 11	0,11	Sterilization

Essential glass filter information

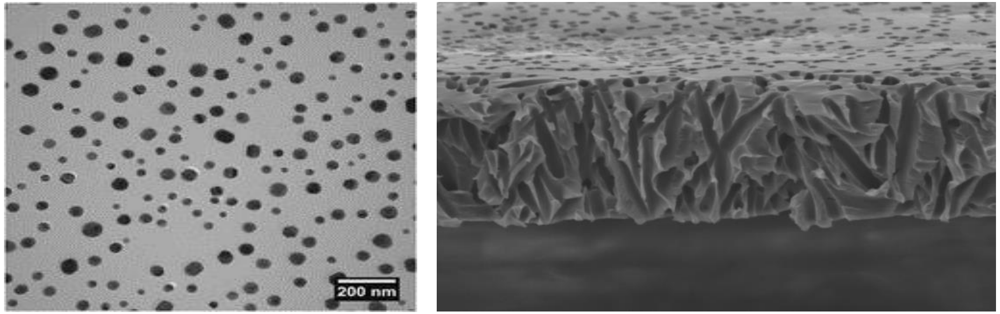
Type	Pore size (μm)	Application
G 00	200-500	refining solutions
G 0	150-200	refining solutions
G1	90-150	refining brews, infusions
G2	40-90	refining brews, infusions, syrups
G3	15-40	filtration of alcoholic and aqueous solutions
G4	3-13	filtration of eye drops
G5	1,0-1,5	sterilizing filtration of injection solutions

Membrane filters

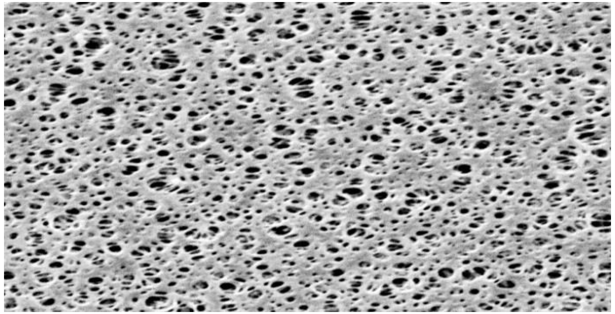


Typical pore sizes for membranes used in reverse osmosis, nanofiltration, ultrafiltration, and microfiltration.

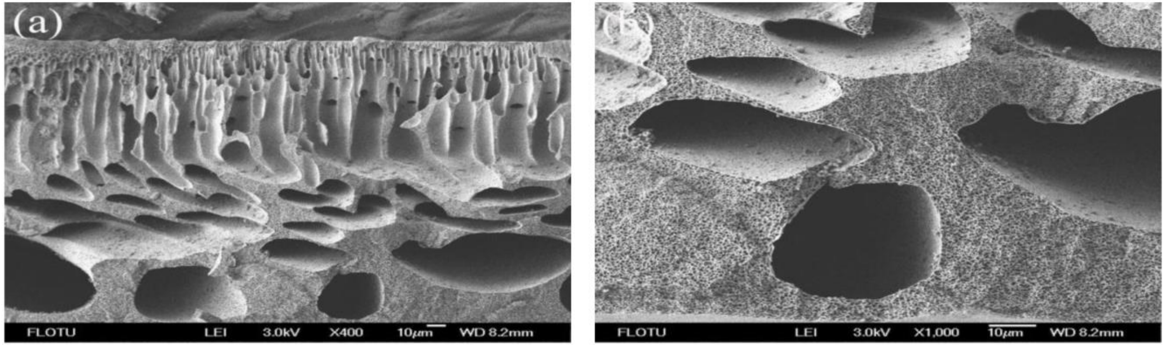
Membrane filters



SEM of Polycarbonate membrane

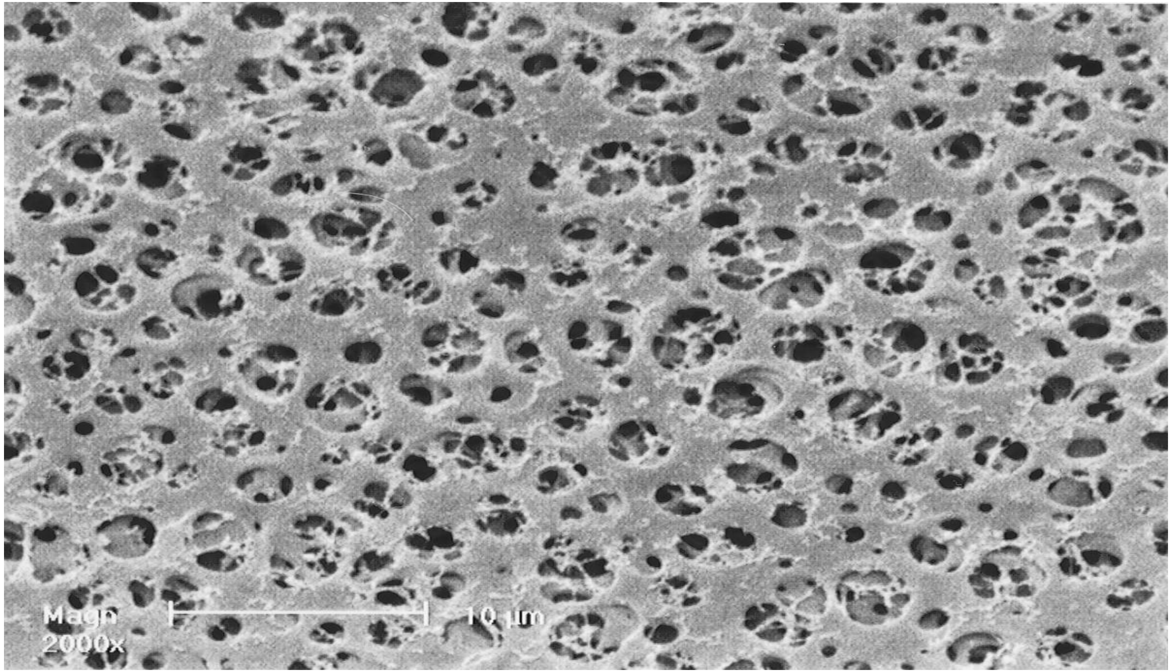


SEM of Cellulose (Polyethersulfone) membrane



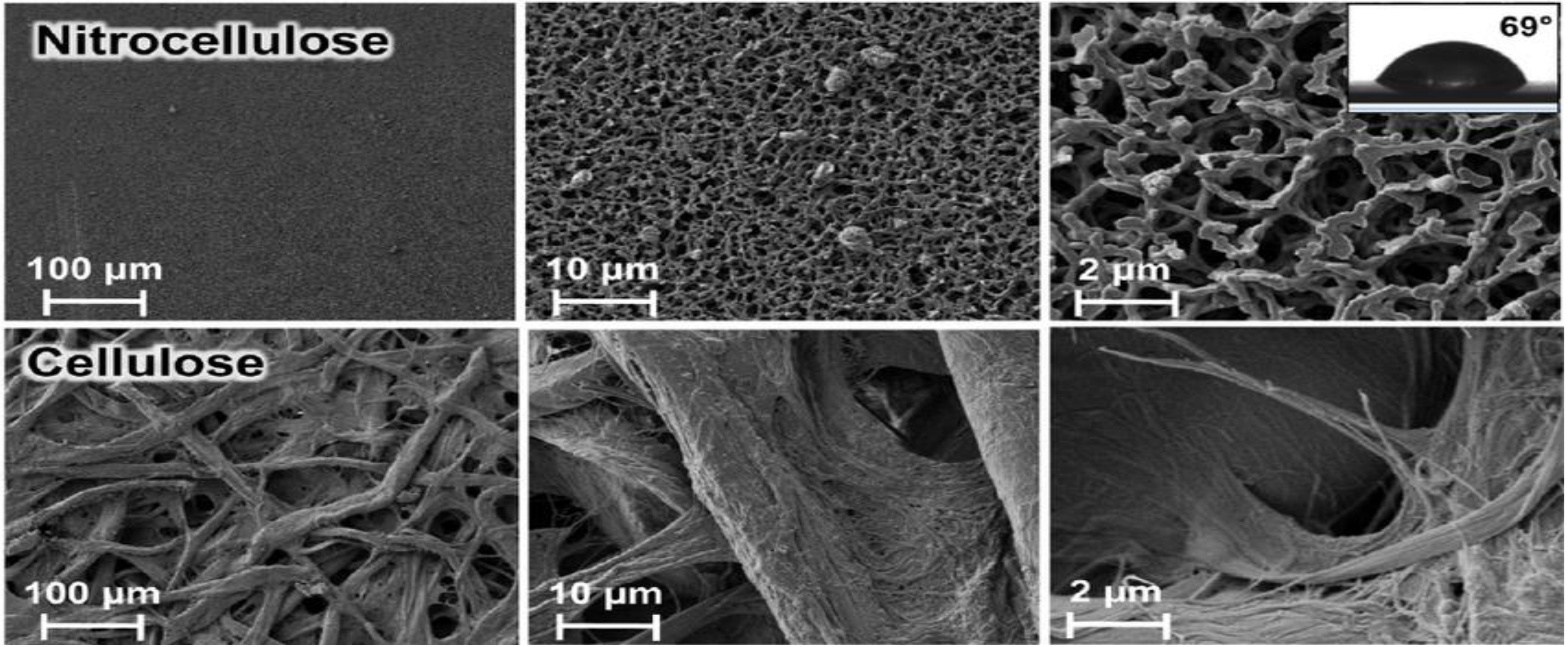
Cross section of Polyphenylsulfone (PPSU) ultrafiltration membrane

Membrane filters



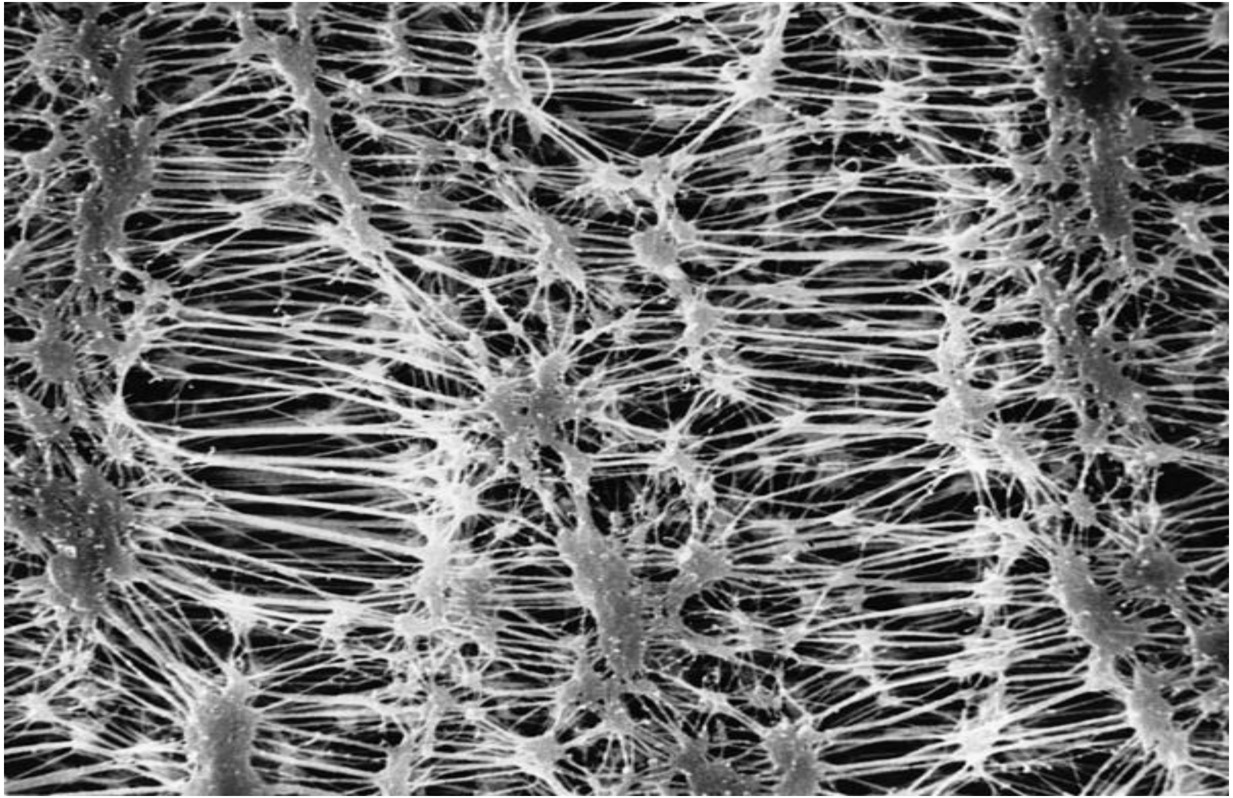
SEM of Cellulose acetate membrane

Membrane filters



SEM of Nitrocellulose and Cellulose membrane

Membrane filters

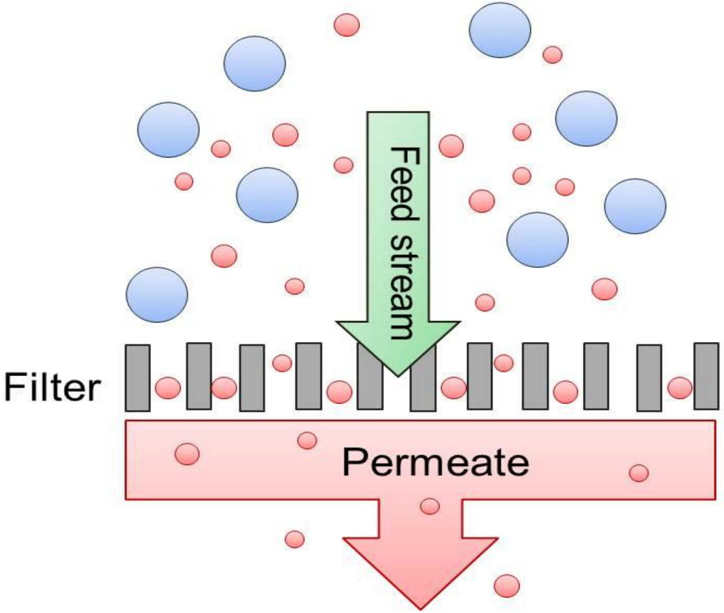


SEM of Fluoropore membrane

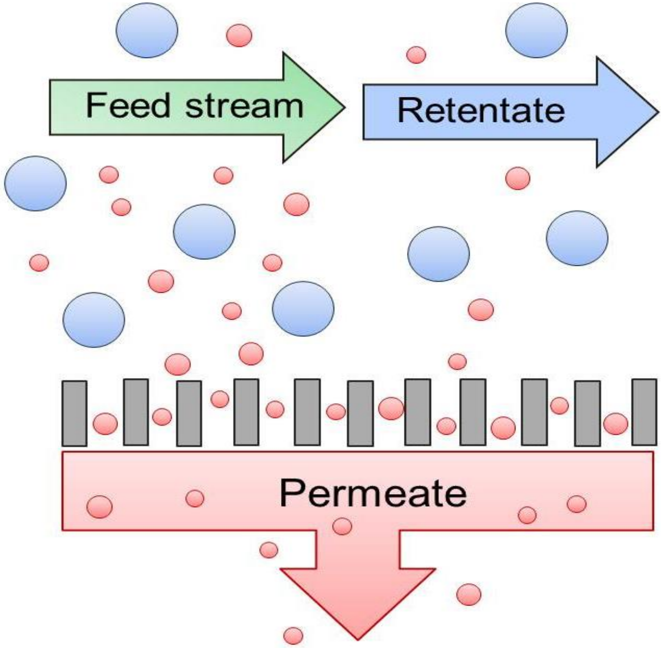
Membrane filters

Type	Manufacturer
cellulose ester	Millipore
nylon	Duralon
cellulose ester and nylon	Mikroweb
cellulose acetate	Celolate
teflon	Mitex
polyvinyl derivatives	Polyvic

Main types of surface filtration



Dead end filtration



Cross flow filtration

Membrane filters

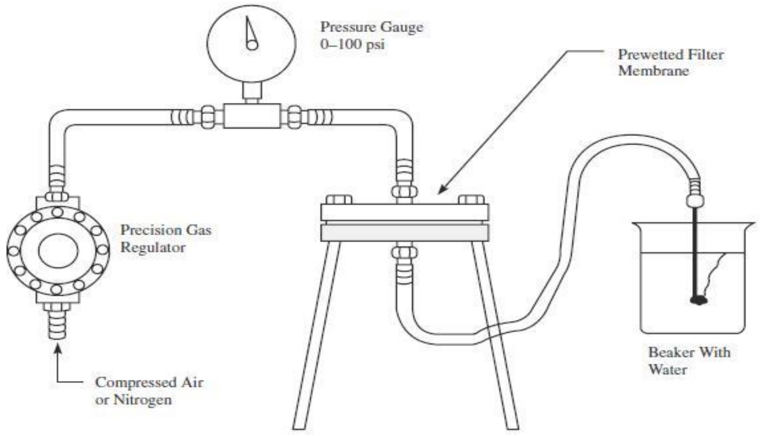
Bubble Point Test

The bubble point measurement determines the **pore size of the filter membrane**, i.e., the larger the pore the lower the bubble point pressure.

At a certain pressure level, liquid will be forced first from the set of largest pore, in keeping with the inverse relationship of the

- *applied air pressure (P) and*
- *the diameter of the pore (d)*
- *γ is the surface tension,*
- *ϑ=wetting angle:*

$$P = \frac{4\gamma \cos\Theta}{d}$$



The bubble point test can be associated with the result of the **bacterial retention** test.

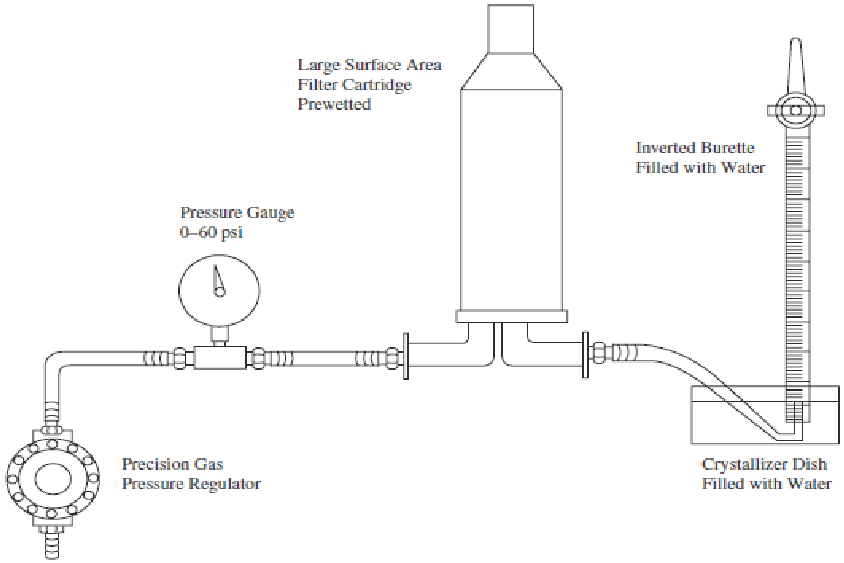
Membrane filters

Diffusion test / diffusive flow test

At a pressure approximately 80% of the minimum bubble point, the gas which diffuses through the membrane is measured to determine a **filter's integrity**.

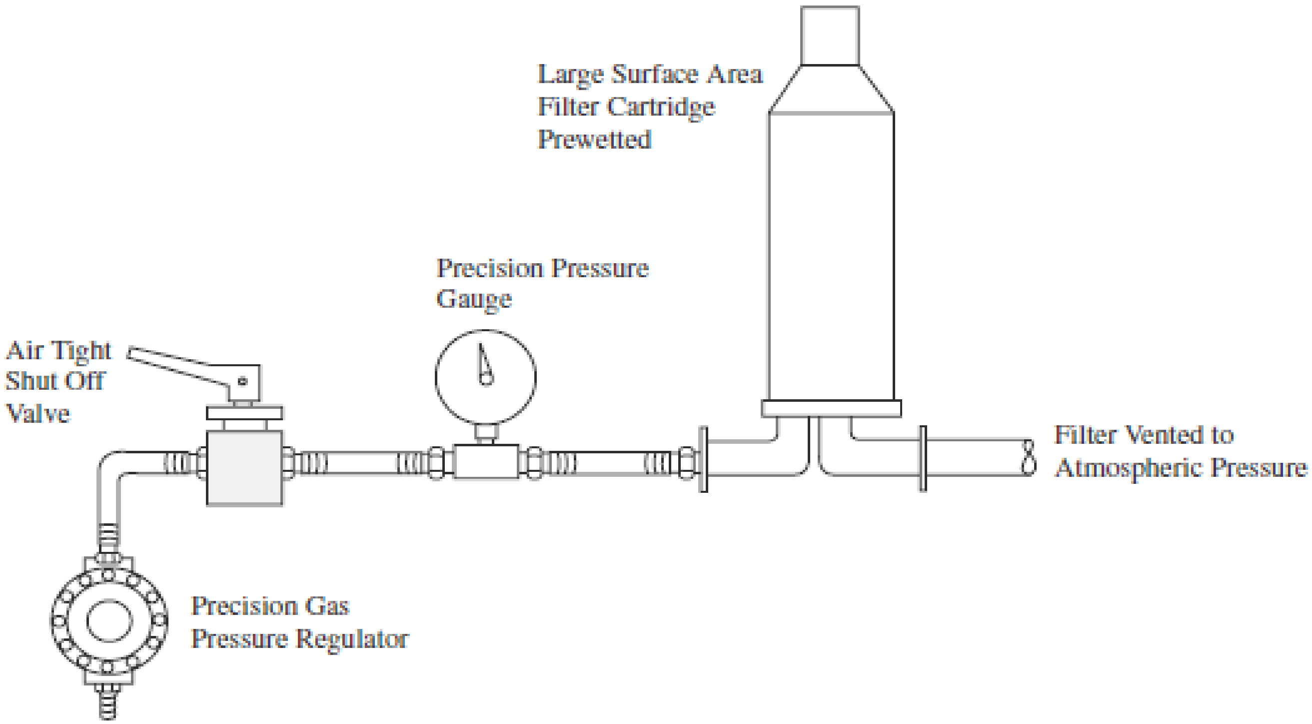
The flow of gas is very low in small area filters, but it is significant in large area filters.

Maximum diffusional flow specifications have been determined for specific membranes and devices and are used to predict bacterial retention test results.



Membrane filters

Pressure Hold Test



Filtration

application

Solutions, syrups: paper, textiles, glass filter

Eye drops: G5 sintered glass filter 1,0- 1,5 µm → suspended contaminants
Membrane filters (elimination of microorganisms)
0,20 µm → water based solution
0,45 µm → oil (viscous) based solution

Injections, infusion: prefiltering→ suspended contaminants
~ hard porcelain, single- and multi-layer filters
endfiltering→ glass, single- and multi-layer filters, membrane filters
i.e. G5 sintered glass filter

De-microbial filters !!!
”those preparations, where terminal sterilization is not possible....
... 0,20 µm membranefilter (see OGYÉI homepage)

Reverse osmosis
preparation of „aqua purificata”

The water is separated from the dissolved substances (forced through a semipermeable membrane).

The larger molecules cannot penetrate trough the membrane pores (metal complexes, organic molecules).

Reverse osmosis
preparation of „aqua purificata”

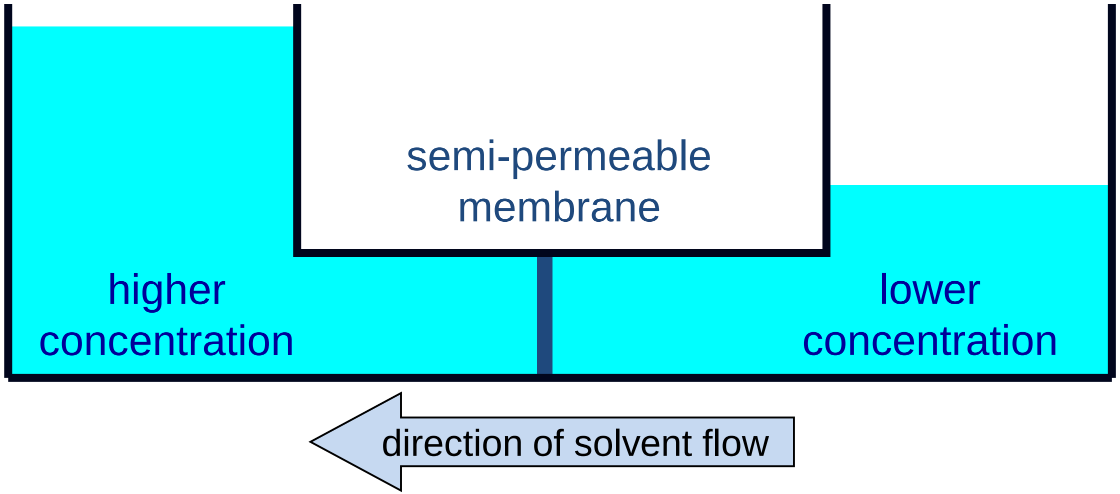
The membrane material is usually **polyamide** or **cellulose-acetate**.

The polyamides can be applied in a large pH-range (2-11).

Carbon and metalic filter’s pore size is larger, 10-100 µm.

Osmosis

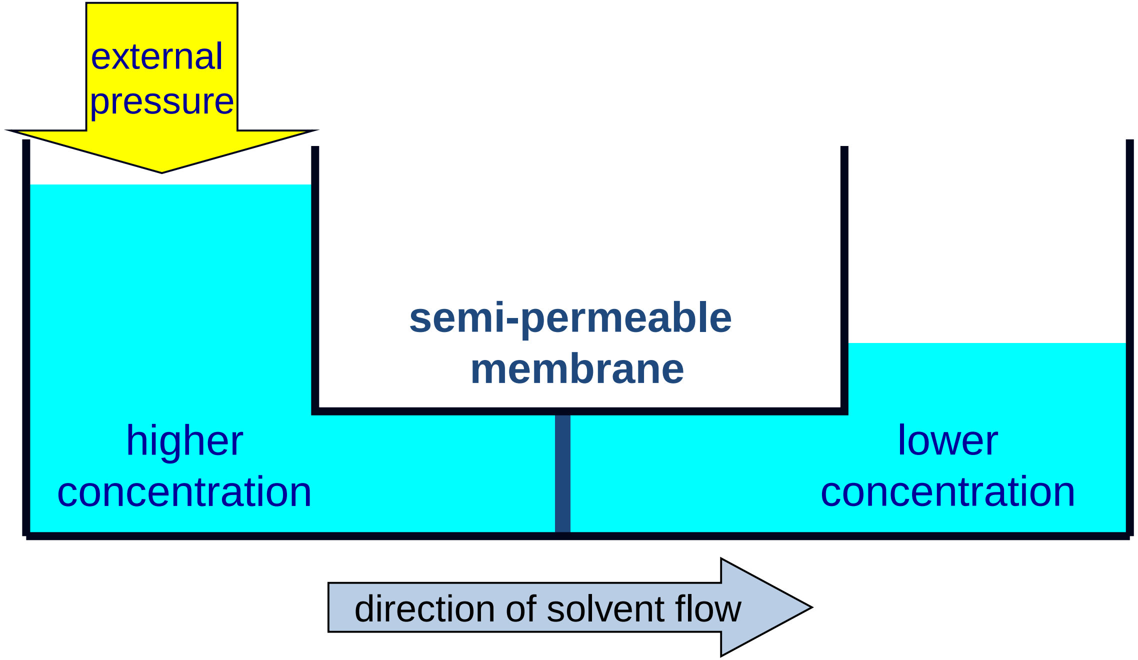
Osmosis is the movement of solvent molecules through a selectively permeable membrane into a region of higher solute concentration, aiming to equalize the solute concentrations on the two sides



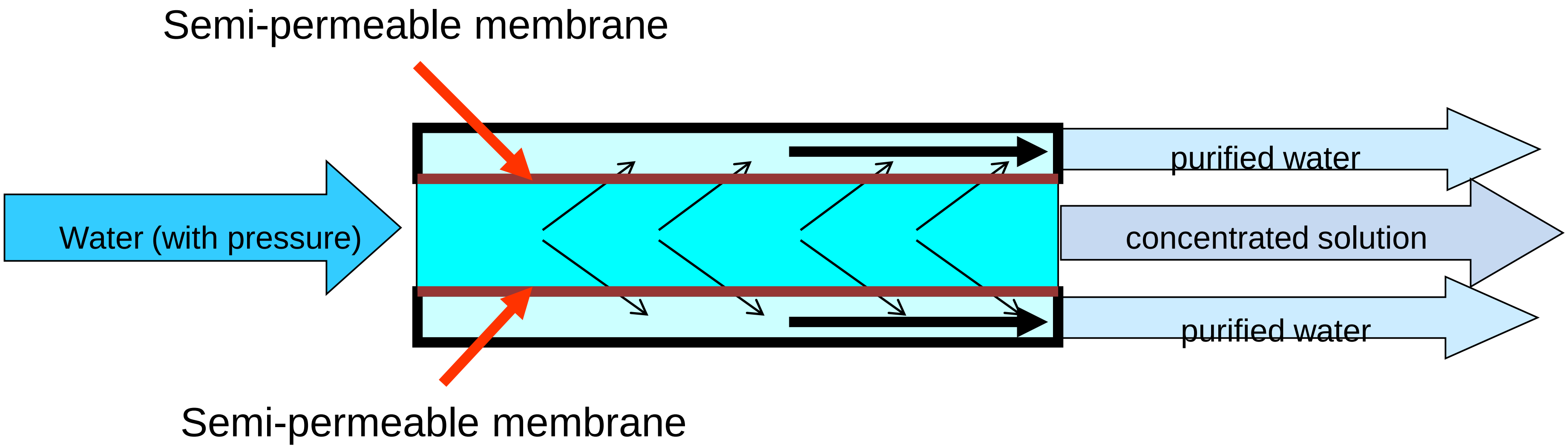
Osmosis

Reverse osmosis

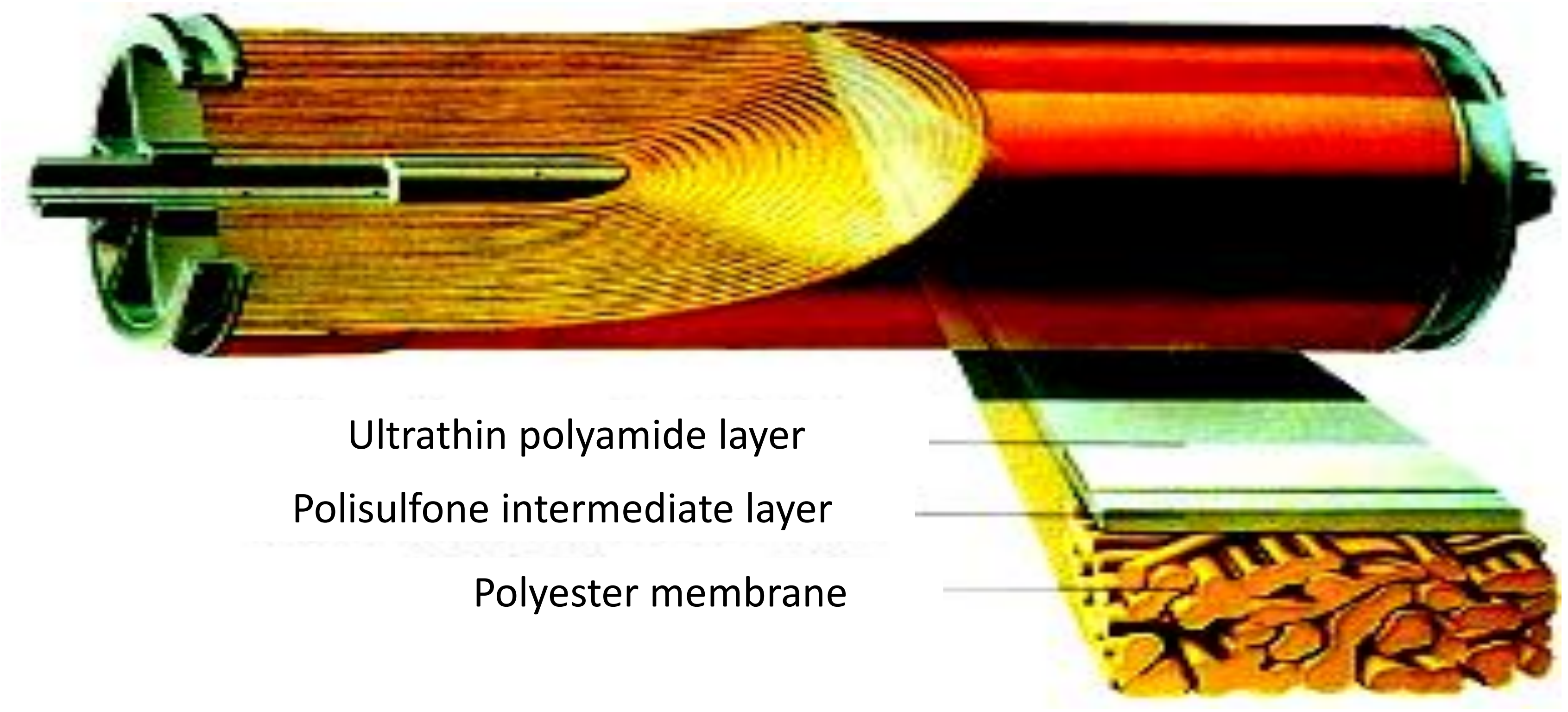
The direction of solvent flow can be reversed by external pressure.



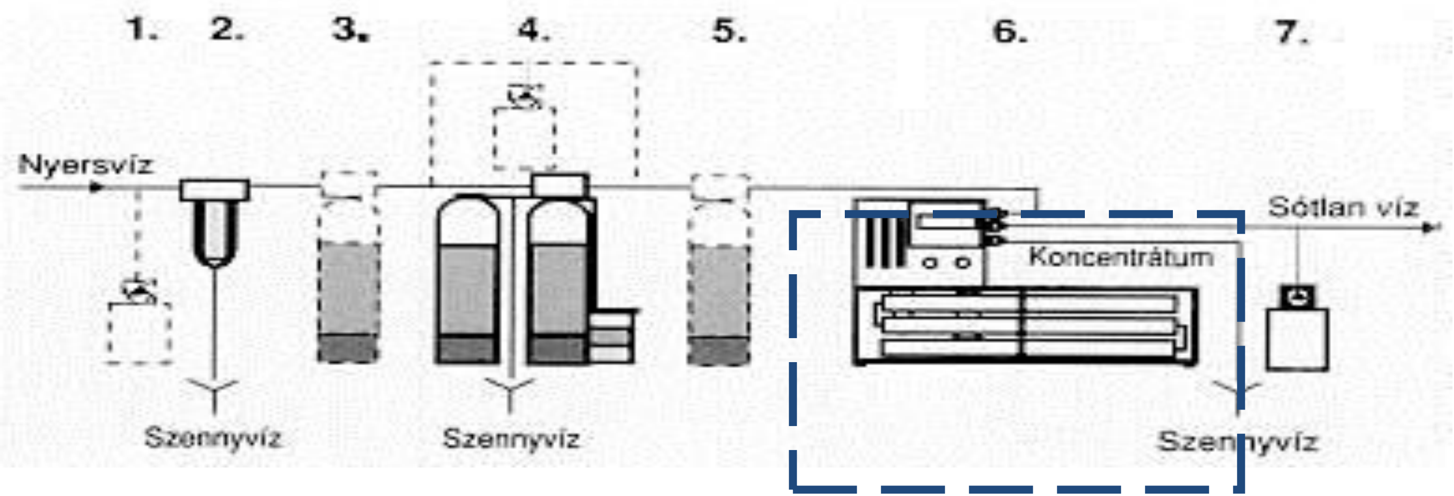
Reverse osmosis



Reverse osmosis



Reverse osmosis



1. Chemical feeder, inhibation of biological contamination
2. Suspended particles filtration, prefiltration
3. Iron removal equipment
4. Twin-column water softener – alternative: chemical feeder
5. Activated charcoal filter with high content of organic matters and free chlorine
6. *Reverse osmosis (the equipment)*
7. Chemical feeder for water conditioning

BASIC OPERATIONS

SEDIMENTATION

Sedimentation

Sedimentation is an **operation**,

where the phases of the disperse system (suspension) are separated

in a **density-dependent** process.

Sedimentation

Stokes equation *(low concentrated suspension)*

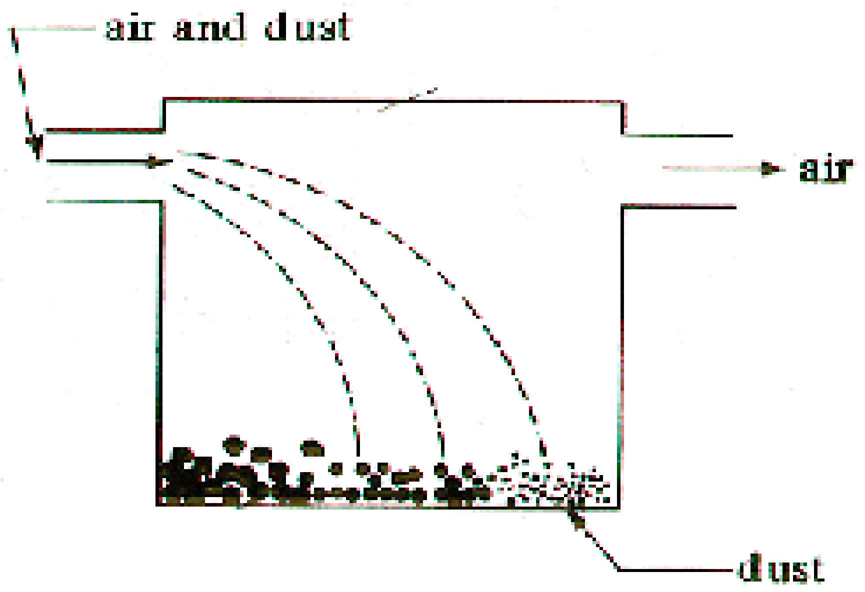
$$v = \frac{2r^2(\varsigma_1 - \varsigma_2)g}{9\eta}$$

v = *sedimentation speed*
r = *radius of the particle*
ς_1 = *density of the dispersed phase*
ς_2 = *density of the dispersion media*
η = *viscosity of the media*
g = *gravity force*

Sedimentation

Separator container/ dust chamber

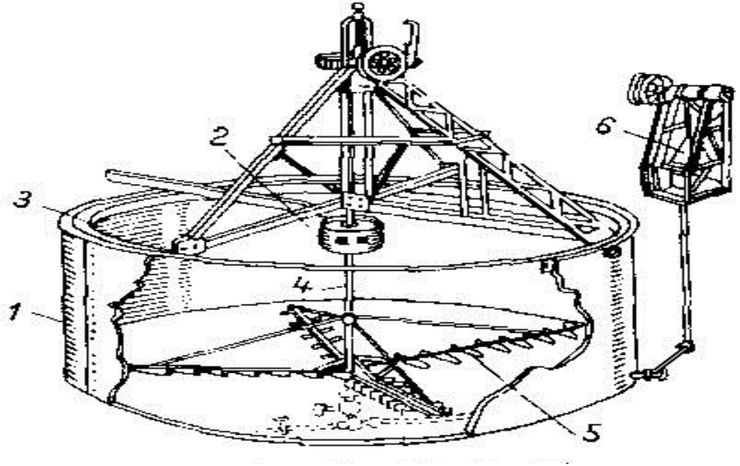
Separation of solid particles, powders, granules by air flow



Sedimentation

Dorr-type sedimentation box

Slow stirring, continuous removal of the sediment



1. container
2. feeder
3. drainage channel
4. mixing shaft
5. mixing blades
6. mud/sediment pump

BASIC OPERATIONS

CENTRIFUGATION

Centrifugation

Centrifugation is an **operation**,

where the phases of the dispers system (suspension) are separated

in a **g-force** elevated environment.

Centrifugation

Application

- Separation of non-miscible liquids
- Separation of suspended solid substances from the solvent
- Remove excess fluid

Theoretical basis of centrifugation

$$F_c = \frac{mv^2}{r}$$

$$v = r\omega$$

$$F_c = mr\omega^2$$

v	=	*peripheral speed*
r	=	*radius of the centrufuge's drum*
ω	=	*angular speed*
g	=	*acceleration due to gravity*
m	=	*mass of the particle*
F_c	=	*centrifugal force*

Theoretical basis of centrifugation

Separation factor of centrifugation (β)

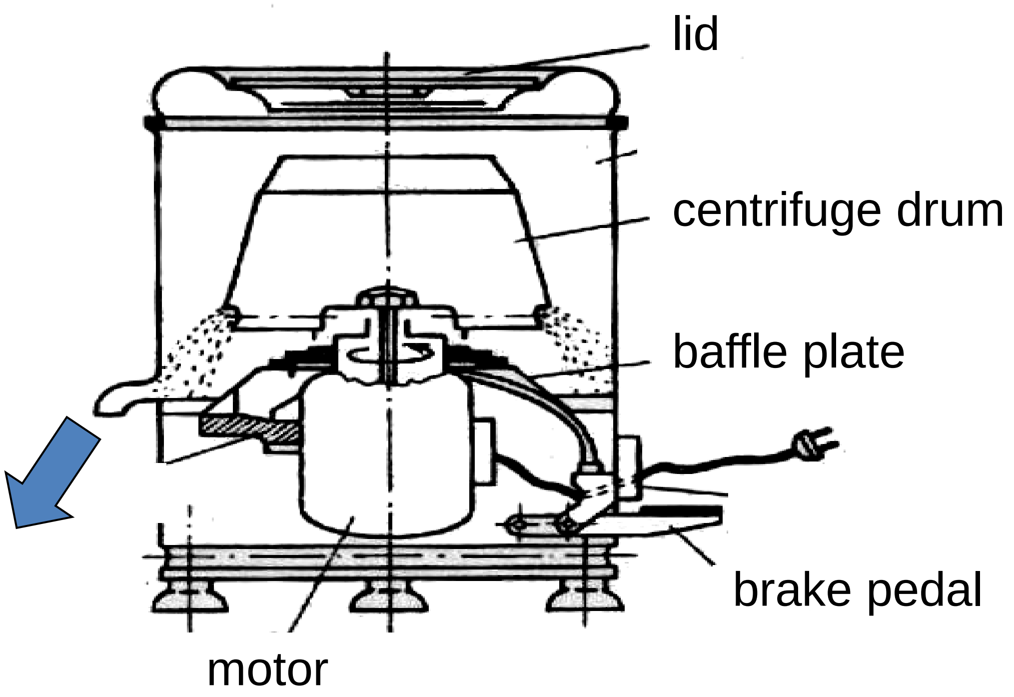
efficacy of the separation,

F_c

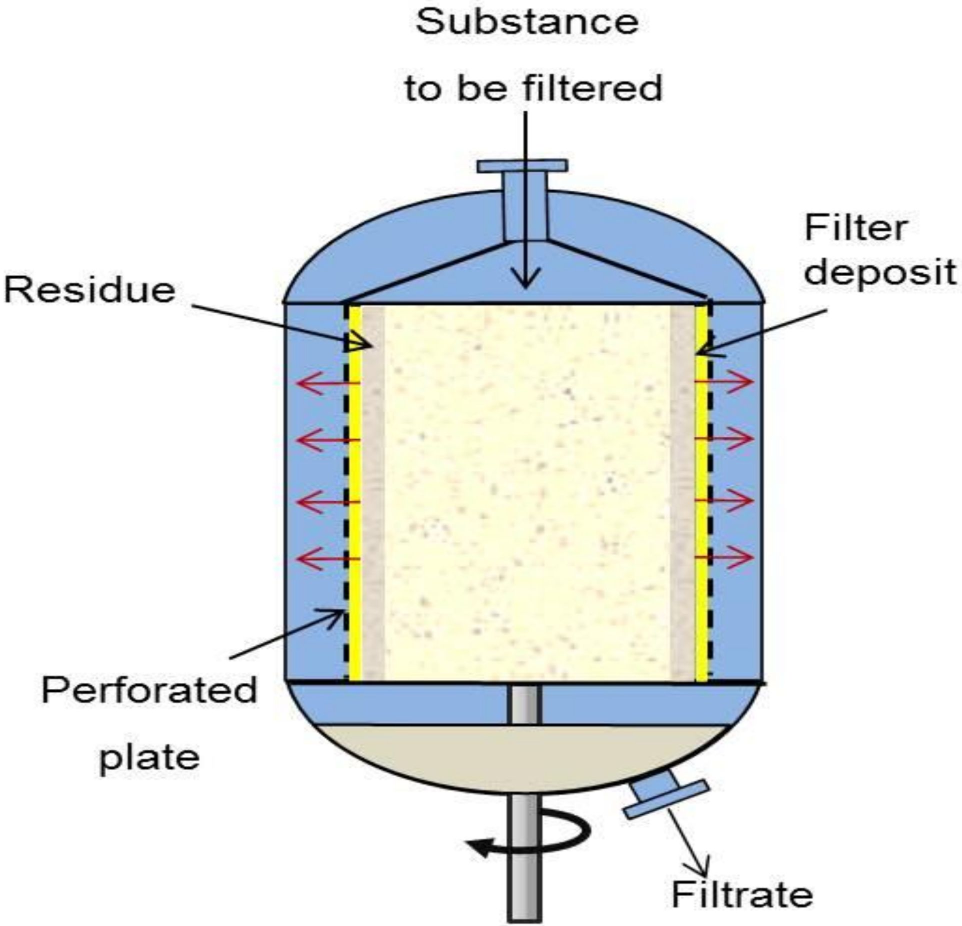
$$\beta = \frac{\frac{mv^2}{r}}{mg} = \frac{v^2}{rg} = \frac{r\omega^2}{g}$$

v	=	*peripheral speed*
r	=	*radius of the centrufuge's drum*
ω	=	*angular speed*
g	=	*acceleration due to gravity*
m	=	*mass of the particle*
F_c	=	*centrifugal force*

Laboratory and industrial centrifuges

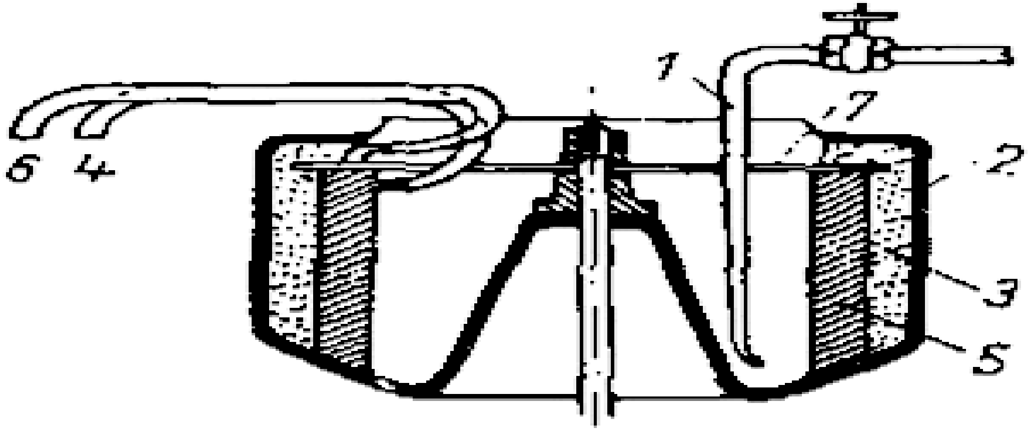


Separator


Substance
to be filtered
Filter
deposit
Residue
Perforated
plate
Filtrate

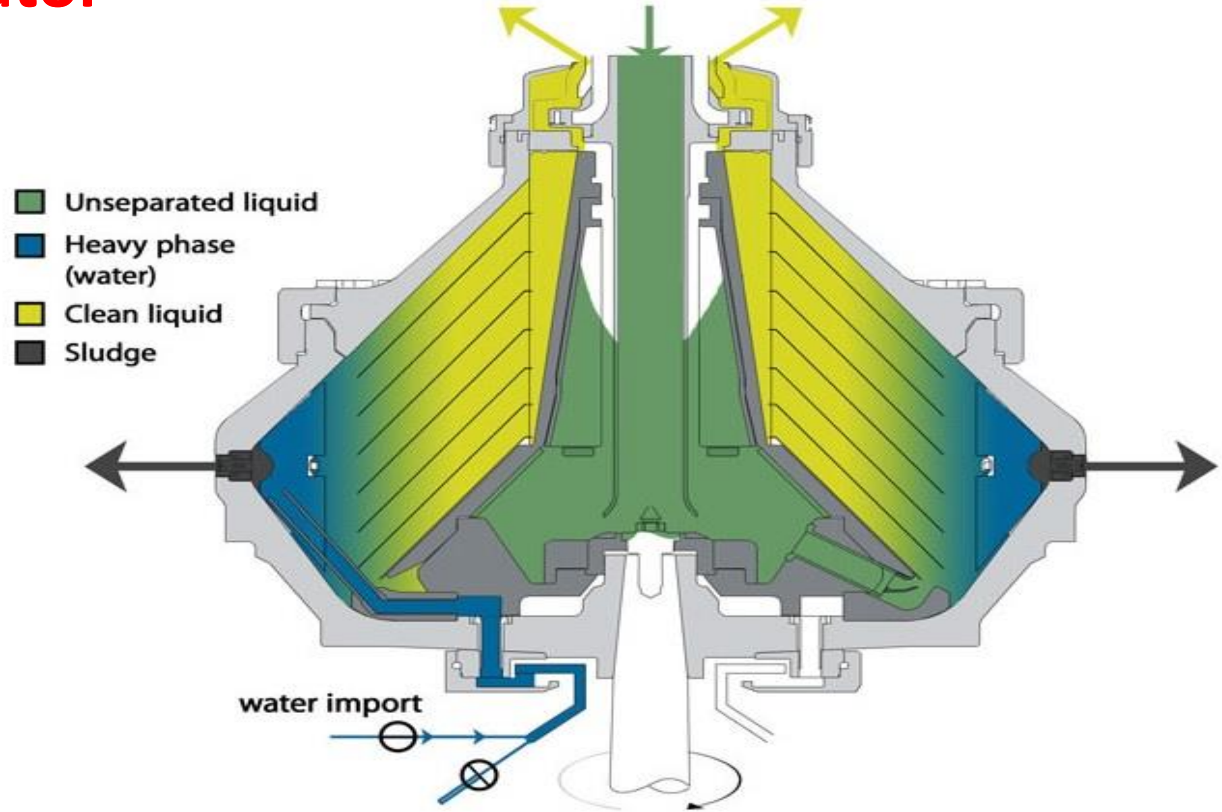
Separator

Separation of emulsions



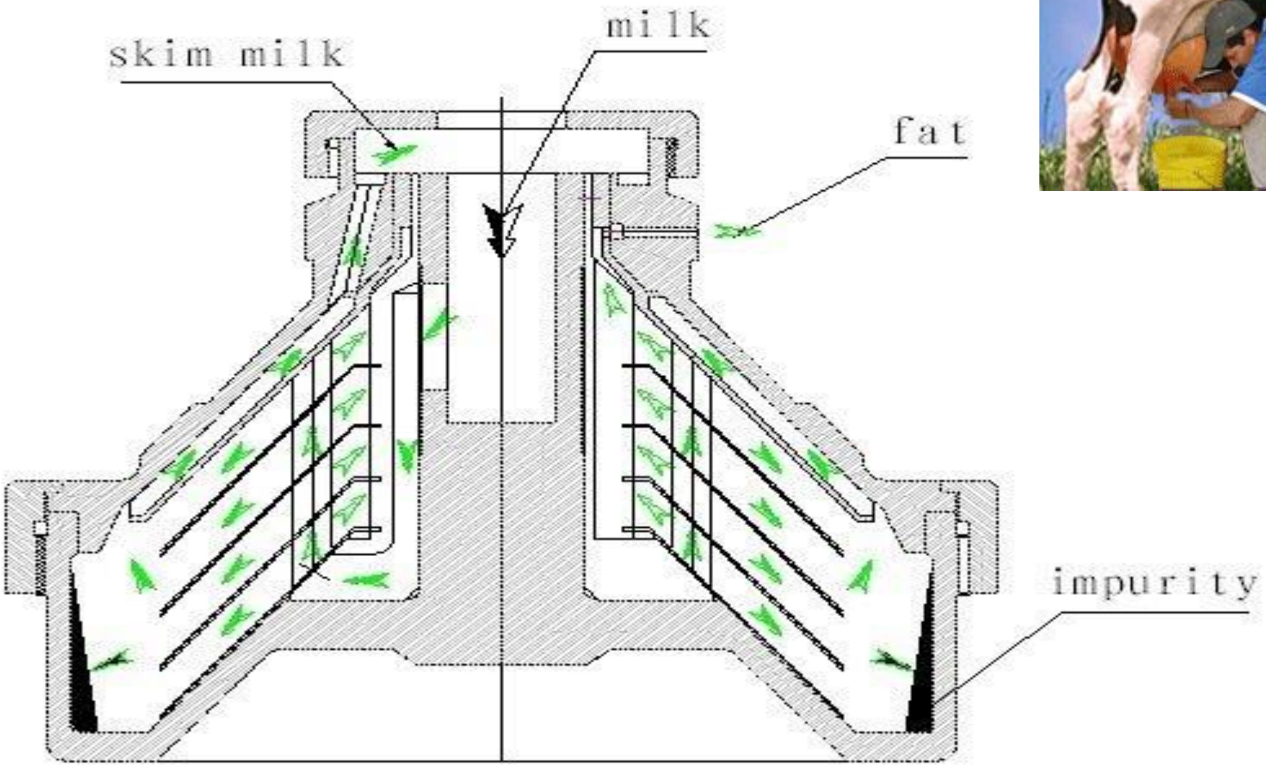
1. emulsion loading tube
2. drum
3. higher density liquid
4. lower density liquid
5. divider wall (filter)
6. drain pipe

Disc-separator



Tapered divider walls are in the drum in which the liquid can separate in thin layer.

Milk-separation



The End