

Extraction

Liquid – Liquid Extraction

Solid – Liquid Extraction

High Pressure Extraction

Lecturer: Thomas Gamse

ao.Univ.Prof.Dipl.-Ing.Dr.techn.

Department of Chemical Engineering and Environmental Technology

Graz University of Technology

Inffeldgasse 25, A-8010 Graz

Tel.: ++43 316 873 7477

Fax: ++43 316 873 7472

email: Thomas.Gamse@TUGraz.at

EXTRACTION

1. Introduction

Extraction is the withdrawing of a active agent or a waste substance from a solid or liquid mixture with a liquid solvent. The solvent is not or only partial miscible with the solid or the liquid. By intensive contact the active agent transfers from the solid or liquid mixture (raffinate) into the solvent (extract). After mixing the two phases are separated which happens either by gravity or centrifugal forces.

For recovery of the solvent and to get the active agent in pure form a further separation process is necessary (rectification or re-extraction)

Depending on the phases following types of extraction exist:

- Solid – liquid extraction
- Liquid – liquid extraction

The gas – liquid extraction is called absorption.

The main area of extraction is for hydrometallic processes, for pharmaceutical industry (producing active agents), for petroleum industry (production of monomers and aromates) and for cleaning of waste water to separate solved compounds.

1.1. Selection of the solvent

The solvent for extraction has to withdraw the active agent from a mixture.

- selectivity: Only the active agent has to be extracted and no further substances which means that a high selectivity is required.
- capacity: To reduce the amount of necessary solvent the capacity of the solvent has to be high.
- miscibility: To achieve simple regeneration of the solvent the miscibility of solvent and primary solvent has to be low.
- difference in density: After extraction the two phases have to be separated in a separator and for this a high difference in density is positive.
- optimal surface tension: σ low \rightarrow low amount of energy for dispersing required; if surface tension < 1 mN/m stable emulsions are produced. $\sigma >$

50 mN/m -> high amount of energy for dispersing and high tendency to coalesce

- recovery: The solvent has to be separated from the extract phase easily to produce solvent free active agents.
- corrosion: If the solvent is corrosive prices for construction increase
- low price
- no or low toxicity
- flame temperature: 25 °C higher than operating temperature
- vapour pressure: To prevent loss of solvent by evaporation a low vapour pressure at operating temperature is required.
- viscosity: A low viscosity of the solvent leads to low pressure drop and good heat and mass transfer.
- chemical and thermal stability

1.2. Regeneration of the solvent

For all extraction processes the regeneration by further separation processes is necessary. By this way pure products are produced and the solvent can be recycled in the extraction process. In many cases the regeneration step is the most cost intensive part of the whole process.

Following possibilities for separating of the solvent are available:

- Rectification: The most common method
- Evaporation: The evaporation of the solvent is used if the active agent is very high volatile. The solvent should have a low boiling temperature and a low heat of evaporation.
- Crystallisation: Cooling the solvent results in crossing the solubility and the active agent falls out and can be separated by mechanically separation processes.
- Extraction: A further extraction step with another solvent can be used to separate the active agent from the first solvent. But the by this way produced extract has to be separated once again.

2. Extraction apparatus

2.1. Specifications

To transfer high amounts of active agent from the starting phase into the extract phase the extraction apparatus has to fulfil following points:

- Production of a large phase boundary which is achieved in most cases by producing fine droplets and dispersing these droplets in the continuous phase.
- High mass transfer coefficient which is achieved in most cases by high relative velocities between the two phases
- No axial remixing
- Fast and complete phase separation after extraction

2.2. Extractor types

2.2.1. Single step mixer - separator

This is the simplest extraction apparatus (see figure 1) which can be operated discontinuously or continuously. The transfer of the active agent from one phase into the other takes place in the mixer and the separation of the two phases is done in the separator by gravity forces.

Advantages: high efficiency per step, high operating area, low apparatus height, insensible for suspended substances

Disadvantages: high ground area necessary, high solvent costs (because of high volumes), high energy and regulating effort

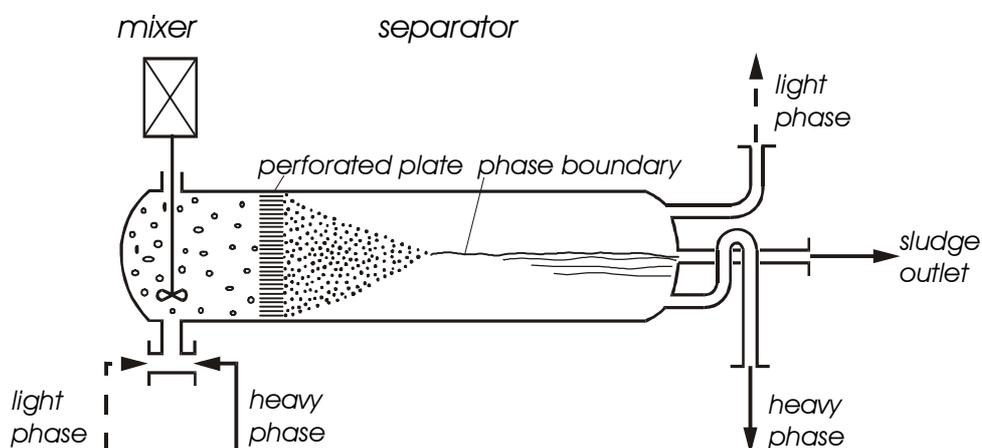


figure 1: single step mixer settler

2.2.2. Mixer – settler - cascade

Adding more mixer – settler units in series a cascade is obtained. These units can be operated in counter current, current and cross flow.

2 types:

- For the box type (see figure 2) mixing and settling zones are separated by plates.
- For the tower type (see figure 3) the single steps are one above the other so that less ground area is used.

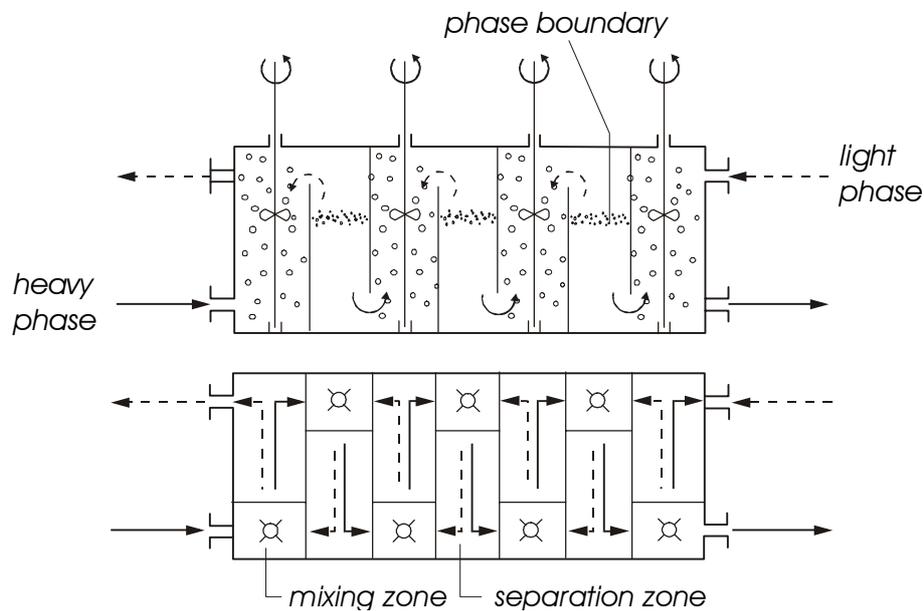


figure 2: box type of a mixer settler cascade.

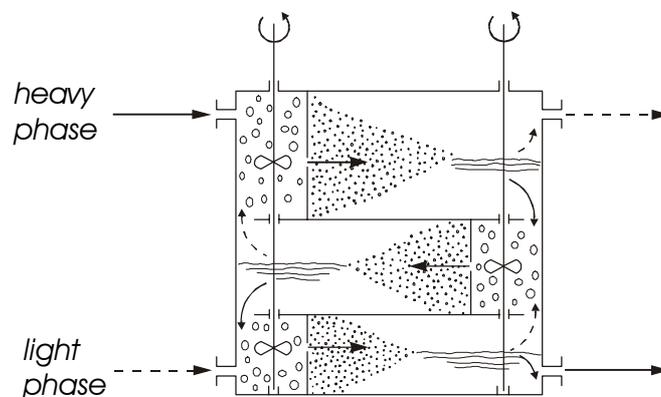


figure 3: tower type of a mixer settler cascade

2.2.3. Centrifugal extractors

Centrifugal extractors exist of a rotating drum where the heavier phase is transported to the periphery and the lighter phase to the centre which means that the centrifugal force is responsible for the counter current flow of the phases.

Advantages: high throughputs, low amounts of solvent and therefore costs, short residence times by fast reaching of the equilibrium

Disadvantages: high costs for investment and operating

The plate extractor (figure 4) exists of a mixing and separating zone where a lot of coned plates are situated. The holes in the plates are for the upwards flowing liquid.

Usage: pharmaceutical industry (extraction of antibiotics), extraction of essences and aroma compounds

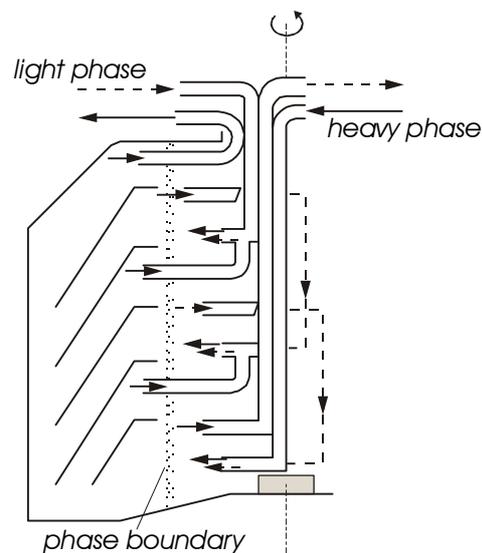


figure 4: plate extractor

2.2.4. Columns without energy input

All columns have a countercurrent flow of the two phases. The reason is the weight force and the difference in density of the phases.

For columns without energy input neither the building of droplets nor the liquid flow is influenced from outside. This is the reason for the negative drop size distribution and the bad renewal of the surface area.

- spraying columns

The spraying column is the simplest type of these columns. Because of the low efficiency and therefore large height this type of column is used only for special purposes. (see figure 5)

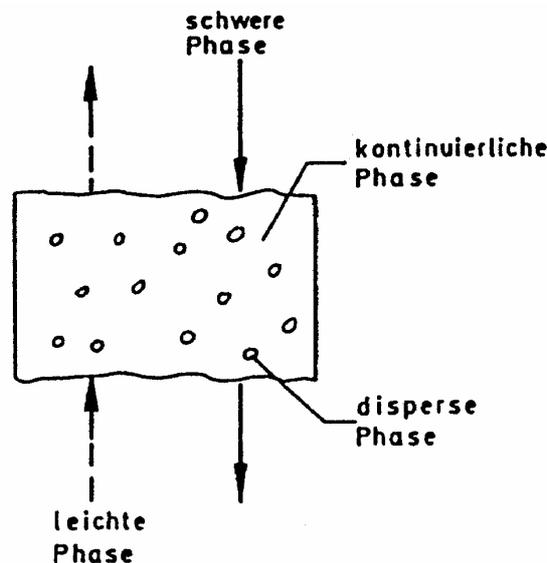


figure 5: spray column

schwere Phase = heavy phase, kontinuierliche Phase = continuous phase,
leichte Phase = light phase

- sieve plate column:

For sieve plate columns the droplets are produced at every plate again. Therefore mass transfer is better (see figure 6). Sieve plate columns work only at high density differences because the pressure drop of the plate has to be overcome.

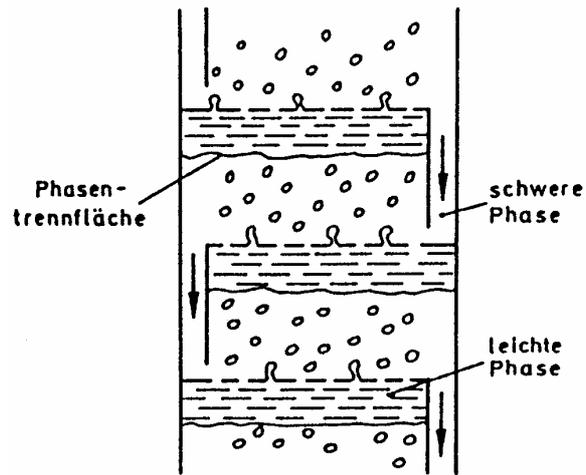


figure 6: sieve plate column

- column with internals

Columns with internals (see figure 7) exist of a columns which is completely filled with internals without distribution between. The distribution of the dispersed phase at the inlet and the wettability of the fillings influence the efficiency of the column.

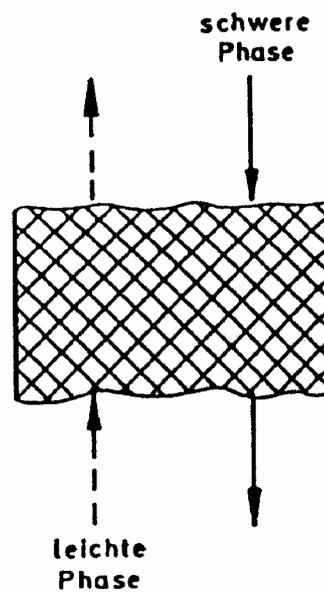


figure 7: column with internals.

2.2.5. Columns with rotating internals

Rotating internals produce a good mass transfer independent of the behaviour of the liquids but the columns become more expensive.

- Scheibel-column

On a vertical shaft mixing and settling zones are mounted (see figure 8). Because of different disadvantages this type of column is not very often used today.

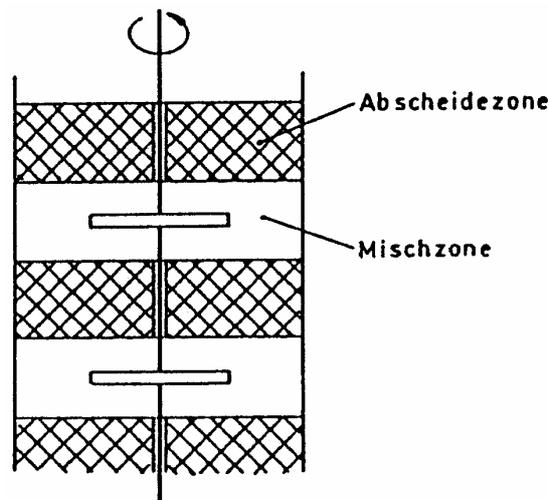


figure 8: Scheibel-column

- Kühni-column

The Kühni-column (see figure 9) is from the principle very similar to the Scheibel-column but instead of the packings stators existing of plates with holes are used.

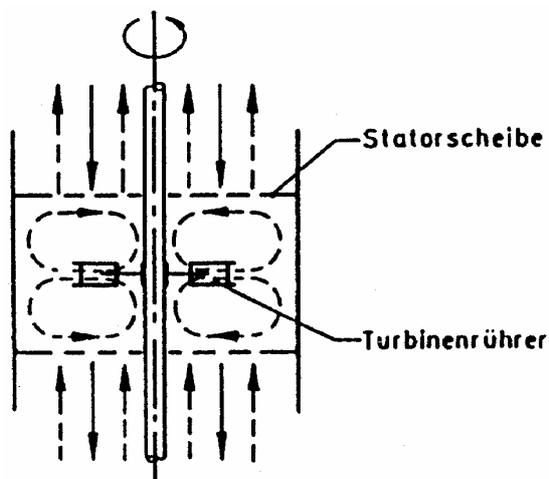


figure 9: Kühni-column.

- RDC [Rotating-Disc-Contactor]

The RDC (see figure 10) is one of the well known type of extractors with rotating internals. On a shaft vertical plates are mounted which are used as mixer. At the wall of the column static rings decrease the axial mixing.

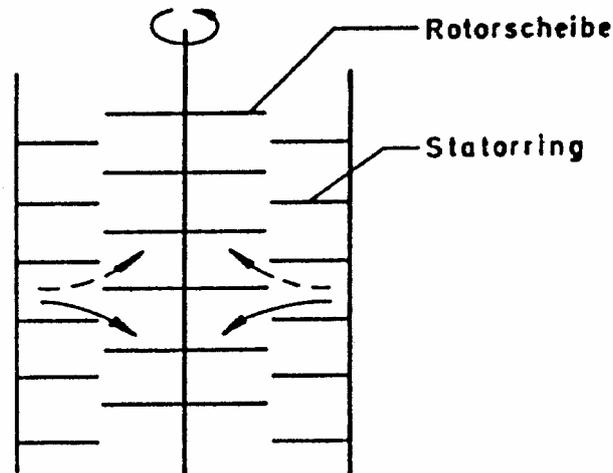


figure 10: RDC

- ARDC [Asymmetrical-Rotating-Disc-Contactor]

The ARDC (see figure 11) is a further development of the RDC. The shaft with the rotating disks is asymmetric to the axis of the column.

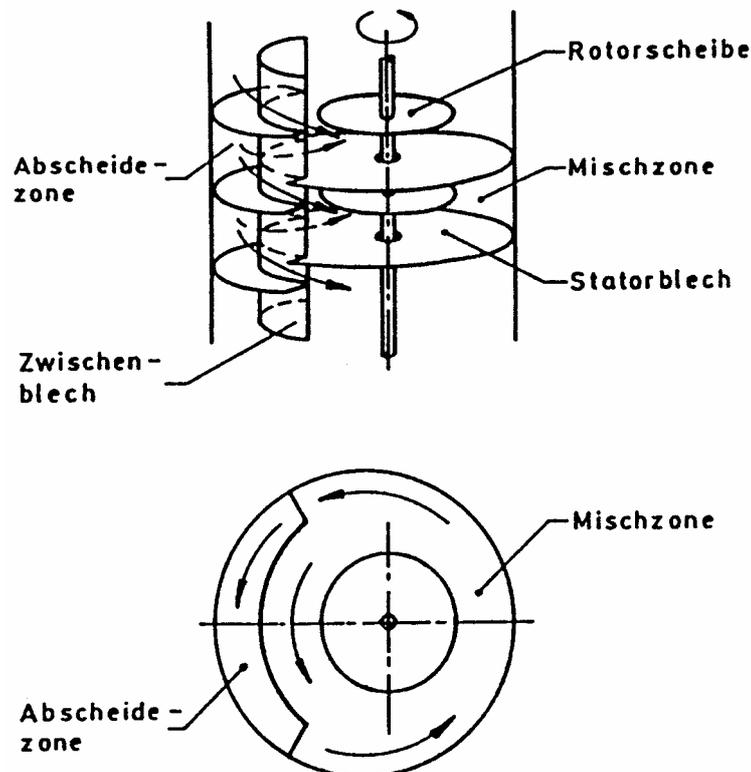


figure 11: ARDC [Asymmetrical-Rotating-Disc-Contactor].

2.2.6. Columns with pulsation

Pulsation increases efficiency dramatically because the surface is renewed and high turbulences at the surface are present.

- Pulsating sieve plate column

The whole area of the column is filled by the plates so that both phases have to pass the holes (see figure 12). This results in always new phase boundary and therefore increase of mass transfer.

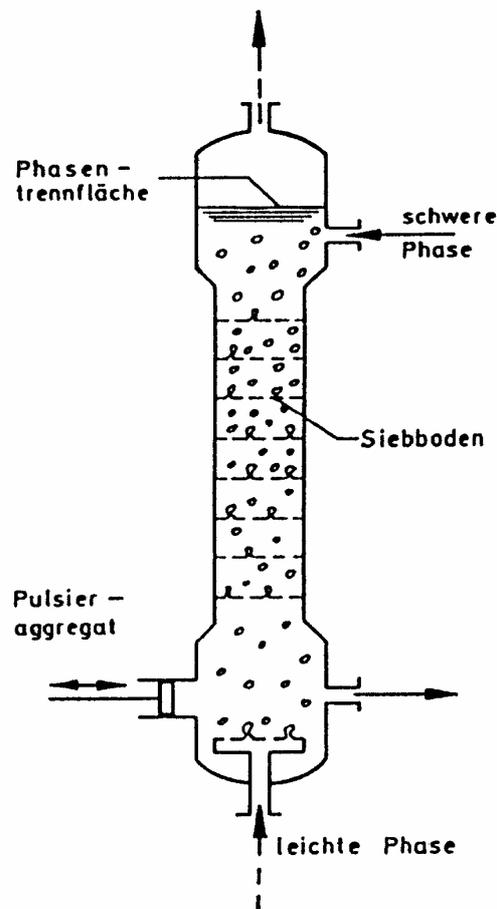


figure 12: pulsating sieve plate column

- Pulsating column with internals

The mass transfer in the pulsating column is better than without pulsation because no channelling of the liquid appears (see figure 13).

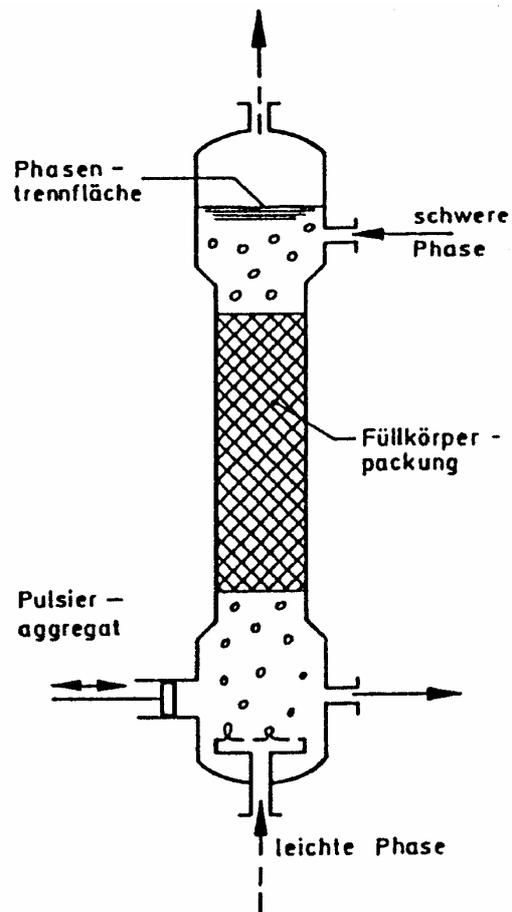


figure 13: pulsating column with internals

- Vibrating plate column

The vibrating plate column (see figure 14), also called Karr-column, has moving sieve plates which cover 50 to 60 % of the free area of the column.

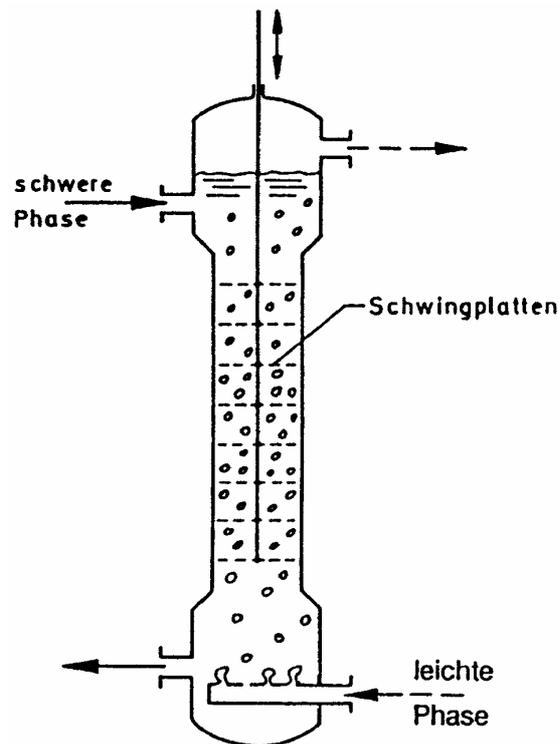


figure 14: vibrating plate column

3. Calculation methods for extraction

3.1. Extraction in step apparatus

If two phases enter an apparatus or a part of an apparatus and are in equilibrium at the outlet this is an ideal step. In reality this equilibrium is not reached and therefore the result has to be corrected with the efficiency.

3.1.1. Single step extraction

One extraction step consists of a mixer and a settler. The feed and the solvent are intensive mixed in the mixer so that the substance to be extracted distributes in the two phases corresponding to the phase equilibrium. The two phases are separated in the settler.

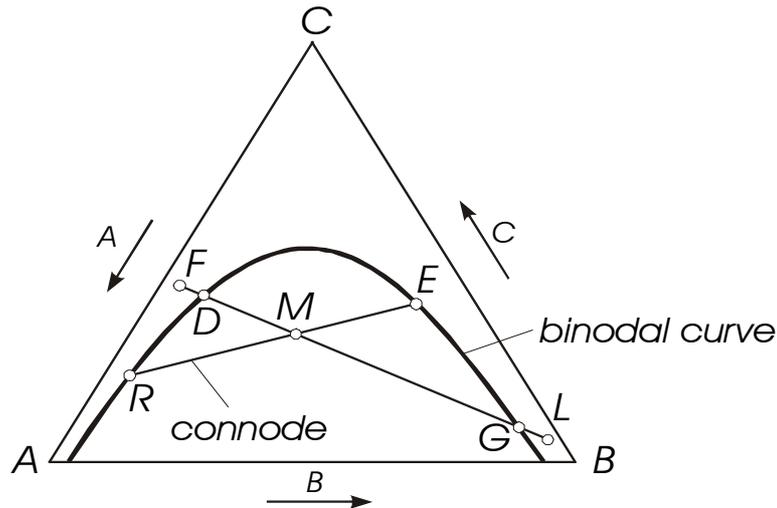


figure 15: single step extraction

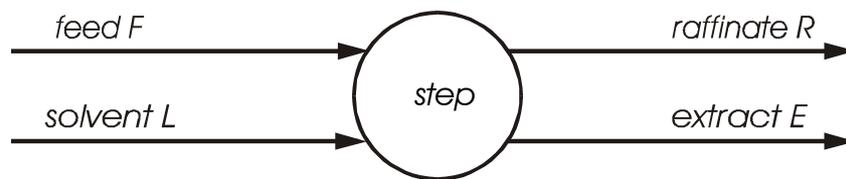


figure 16: single step extraction

The single step extraction can be operated in continuous and discontinuous way.

Determining the efficiency two steps are necessary:

1. Mixer: Production of the mixture M from feed F and solvent L

The position of the mixing point M can be determined graphically with the law of balance or by calculation from the mass balance:

- graphically by law of balance:

$$\frac{\overline{FM}}{\overline{ML}} = \frac{L}{F} \quad (1)$$

- calculating from mass balance:

$$\text{total balance: } F + L = M \quad (2)$$

compound balance for compound A:

$$F \cdot x_{A,F} + L \cdot x_{A,L} = M \cdot x_{A,M} \quad (3)$$

$$x_{A,M} = \frac{F \cdot x_{A,F} + L \cdot x_{A,L}}{F + L} \quad (4)$$

2. Settler: Separation of the mixture M in raffinate R and extract E:

If the position of the mixing point M is fixed the compositions of raffinate R and extract E can be determined by the connode going through the mixing point M.

For the graphical determination again the law of balance is used:

$$\frac{\overline{RM}}{\overline{ME}} = \frac{E}{R} \quad (5)$$

calculation is done by mass balance:

$$E + R = M = F + L \quad (6)$$

$$E \cdot x_{A,E} + R \cdot x_{A,R} = M \cdot x_{A,M} \quad (7)$$

$$E = \frac{x_{A,M} - x_{A,R}}{x_{A,E} - x_{A,R}} \cdot M \quad (8)$$

$$R = \frac{x_{A,E} - x_{A,M}}{x_{A,E} - x_{A,R}} \cdot M \quad (9)$$

From the triangle diagram it is obvious that a separation of the two phases is only possible if the mixing point M lays in the two phase region. The crossing points of the line \overline{FL} with the binodal curve are the extrema for M (minimum and maximum amount of solvent).

If M is equal with point D results the minimum amount of solvent. If this amount of solvent is mixed with feed F only raffinate (with the composition D) and no extract is produced.

$$M_{\min} = \frac{x_{A,F} - x_{A,M_{\min}}}{x_{A,M_{\min}} - x_{A,L}} \cdot F \quad (10)$$

From the law of balance it is obvious that the minimum amount of solvent has to be farthest away from point L.

$$\frac{\overline{FM_{\min}}}{M_{\min} L} = \frac{M_{\min}}{F} \quad (11)$$

If M is equal with point G results the maximum amount of solvent M_{\max} . Only extract and no raffinate is produced.

$$M_{\max} = \frac{x_{A,F} - x_{A,M_{\max}}}{x_{A,M_{\max}} - x_{A,L}} \cdot F \quad (12)$$

or by the law of balance

$$\frac{FM_{\max}}{M_{\max}L} = \frac{M_{\max}}{F} \tag{13}$$

3.1.2. Multi step extraction

3.1.2.1. Multi step extraction with cross flow

This kind of extraction is an extension of the single step extraction because more single step units are combined as given in figure 17.

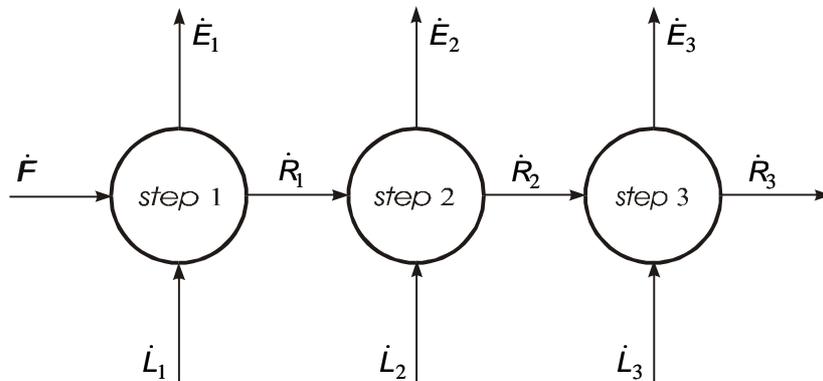


figure 17: multi step extraction with cross flow

For the multi step extraction with cross flow the raffinate of each step is contacted in the following step with pure solvent. The extracts are withdrawn from each step and given to the solvent regeneration. The concentration of compound C in raffinate and extract decreases from step to step.

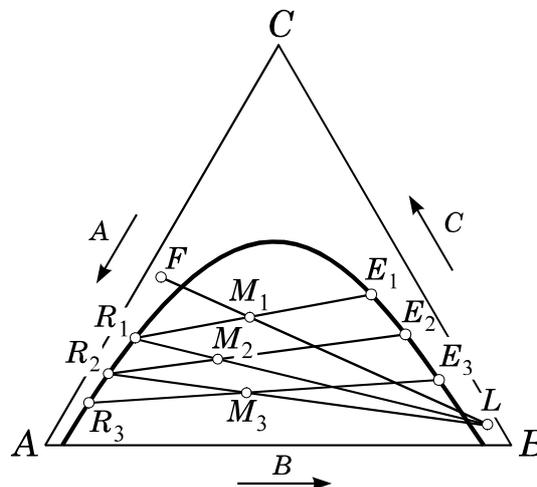


figure 18: multi step extraction with cross flow

If the point of feed F and solvent L are known the first mixing point M_1 can be determined in the same way as for the single step extraction. This mixing point separates in raffinate R_1 and extract E_1 . For the following steps the raffinate is the feed which is contacted with solvent L .

The total extract results from the extract of the single steps:

$$\dot{E} = \sum_{i=1}^n \dot{E}_i \quad (14)$$

The last raffinate concentration (in this case R_3) can also be achieved in a single step extraction. The corresponding mixing point can be constructed as crossing point of \overline{FL} and $\overline{R_3E_3}$. By the law of balance it is obvious that the amount of solvent for the single step extraction is much higher than for the multi step extraction with cross flow.

3.1.2.2. Multi step extraction with counter current flow

The feed and the solvent flow in counter current way through the apparatus. This is a continuous process where feed and solvent enter the apparatus at opposite ends. While raffinate is contacted with pure solvent the extract is contacted with the feed.

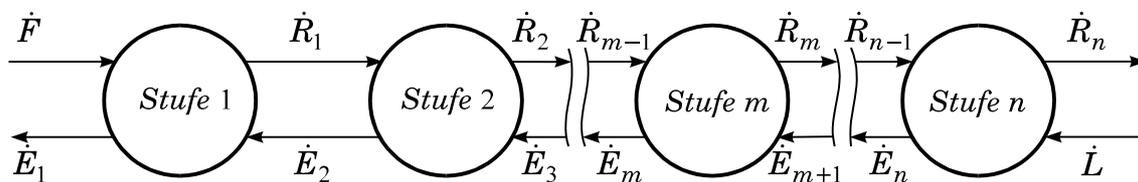


figure 19: multi step extraction with counter current flow

CONSTRUCTION OF THE TRIANGLE DIAGRAM

Basic for the construction are the mass balances. It is obvious that the difference of the mass flows Δ in a section between two steps is constant. The result is that the balance lines cross in one point, the pole point P

total balance:

$$\dot{F} - \dot{E}_1 = \dot{R}_n - \dot{L} = \Delta \quad (15)$$

balance for one step (e.g. m):

$$\dot{R}_{m-1} - \dot{E}_m = \dot{R}_m - \dot{E}_{m+1} = \Delta \quad (16)$$

With Δ as a hypothetical amount of the pole point P results the amount F and the single raffinate as mixing point of P with the corresponding extracts. If L is given and R_n is wanted, with the knowledge of the hypothetical pole point amount results:

$$\dot{R}_n = \dot{P} + \dot{L} \text{ or. } \dot{R}_n = \dot{L} \cdot \frac{\overline{PL}}{\overline{PR}_n}$$

If R_n is given so L can be determined by the law of balance:

$$\dot{L} = \dot{R}_n \cdot \frac{\overline{R_n P}}{\overline{PL}}$$

The position of the pole point P results as crossing point of the lines $\overline{FE_1}$ and $\overline{R_n L}$. In most cases not all four points F , E_1 , R_n and L are given. Normally F , the raffinate concentration R_n and either the extract concentration E_1 or the amount of solvent are given. With the help of the mixing point the missing point can be determined.

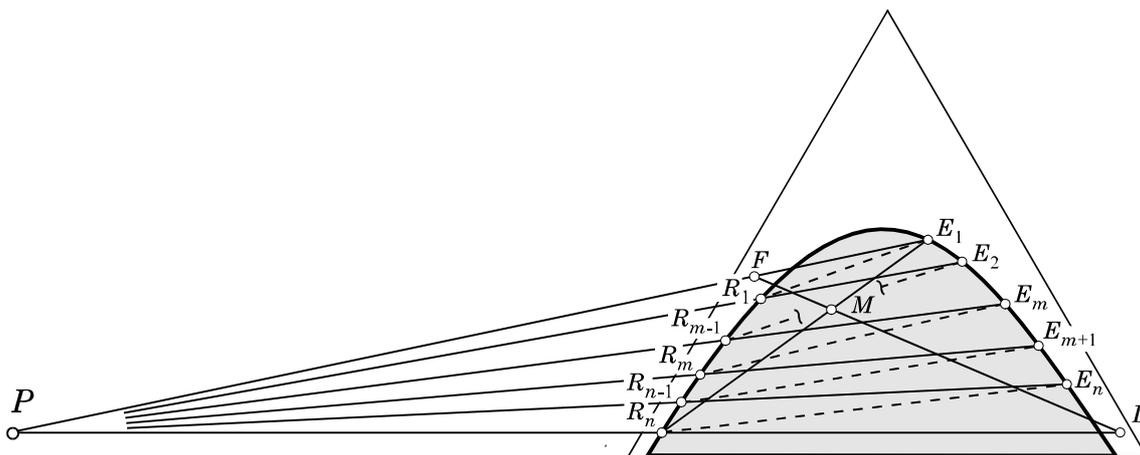


figure 20: multi step extraction with counter current flow.

Phases in equilibrium are combined by a connode and therefore after construction of the pole point the raffinate R_1 according to extract E_1 can be determined. Combining this raffinate R_1 with the pole point last in the extract E_2 of the next step. The line $\overline{PR_1 E_2}$ is a balance line. This construction is repeated until the desired raffinate concentration is reached. By the numbers of raffinate points the number of theoretical steps is determined.

DETERMINATION OF THE MINIMUM AMOUNT OF SOLVENT

In the same way as for other thermal processes the minimum amount of solvent is this amount of solvent which is necessary to achieve a certain separation with maximum consumption (= infinite number of steps).

In general the minimum amount of solvent for the counter current extraction is determined following:

All connodes are extended and crossing points with the line $\overline{LR_n}$ are constructed. This crossing point which is farthest away from point A is the pole point for minimum amount of solvent (see figure 21).

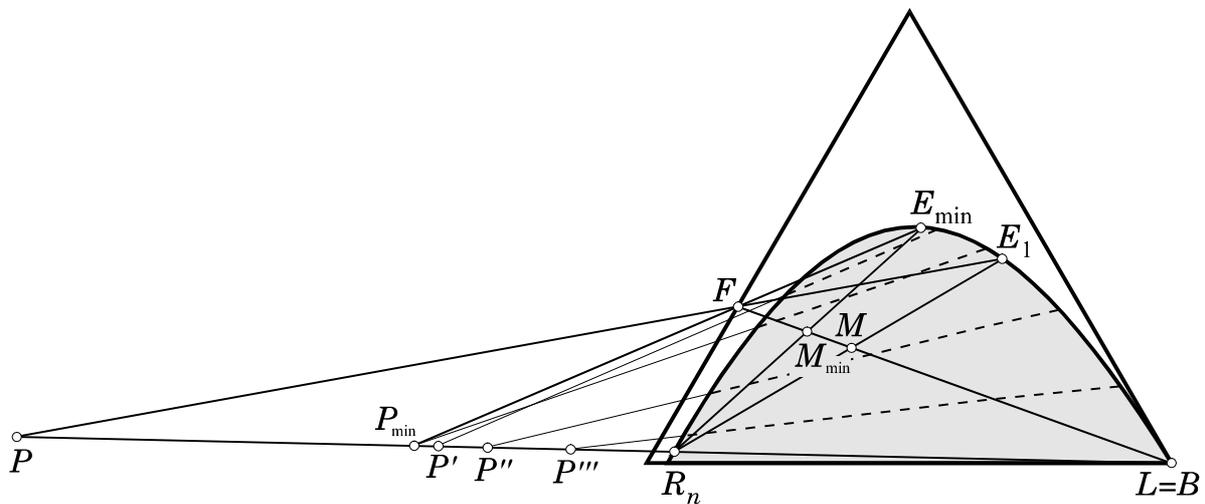


figure 21: determination of minimum amount of solvent for counter current extraction

For the case that the pole point is on the other side the construction is the same as given in figure 22. A large amount of solvent is given (at M_1 respectively P_1). The amount of solvent is the ration between solvent flow and feed flow which can be determined from the triangle diagram:

$$\frac{\dot{L}}{\dot{F}_S} = \frac{\overline{FM}}{\overline{ML}} \tag{17}$$

Reducing the amount of solvent results in moving the mixing point M on the line \overline{FL} in direction of F . With this the extract point E_1 moves on the binodal line upwards so that the pole point P goes farer away until it is infinite. For this case the mixing point is in the position that the lines $\overline{FE_1}$ and $\overline{R_nL}$ are parallel. Further reducing of the amount of solvent brings the pole point to the other side of the triangle diagram and the pole point comes closer.

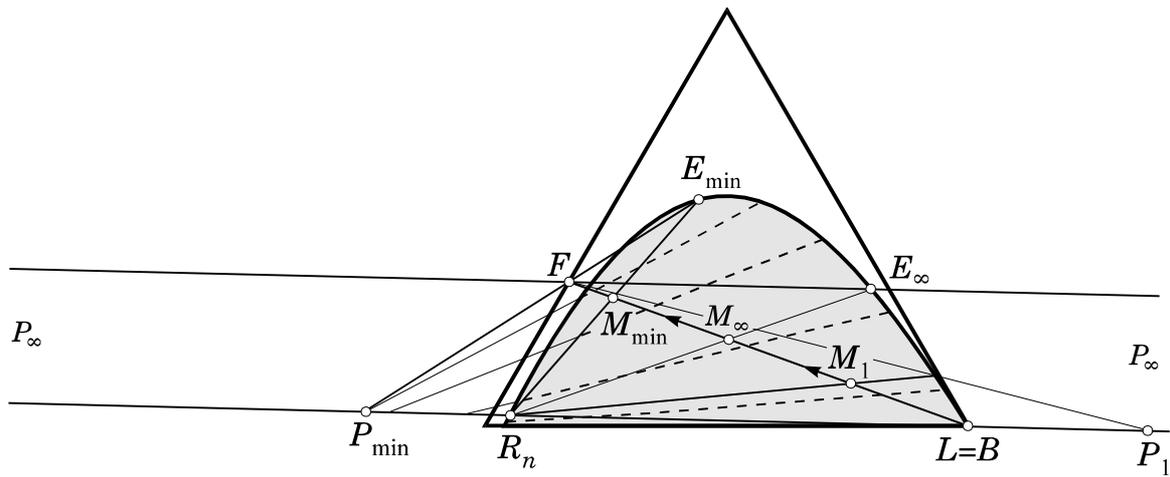


figure 22: minimum amount of solvent with infinite pole point

4. Solid-liquid extraction (Leaching)

4.1. Principles

The principle for the solid-liquid extraction is that the soluble compounds of a solid matter, existing of an inert matrix and the active agent, are extracted by a solvent. The extract can be included in the extraction matter in solid or liquid form. It can be included in cells like oil in oil seeds or as fine dispersion on the solid matter like caffeine in coffee.

Following points are necessary for a economic extraction process:

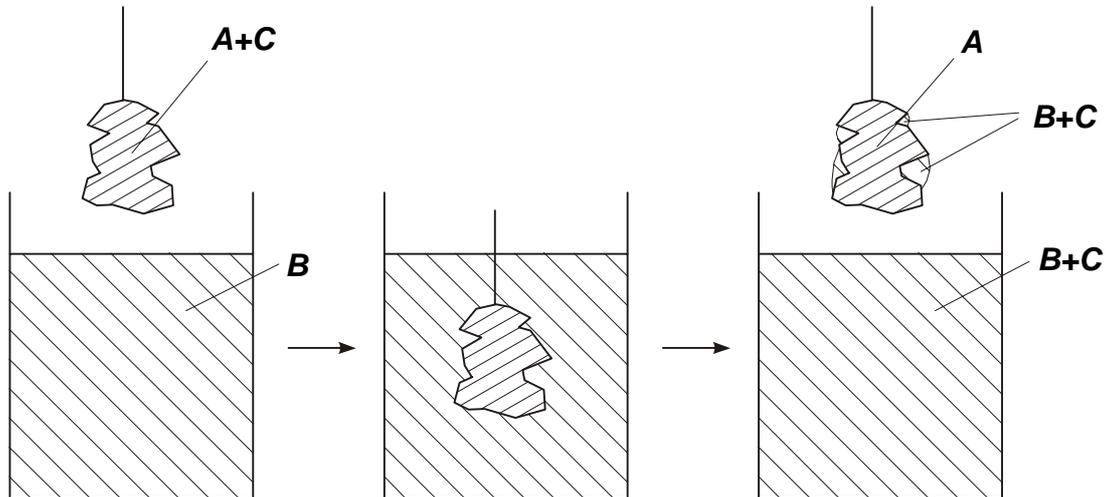
- The extraction matter has to be prepared in this way that the extract can be solved by the solvent in short time. This is achieved by grinding, milling or rolling.
- Only the desired extract has to be solved and extracted. This is achieved by the selectivity of the solvent and the temperature.
- The extract should contain high concentrations of extracted compounds. This is the reason why counter current extraction plants are preferred.
- Separation of the solvent from as well extract solution as extraction residual has to be economically.

A total solid-liquid extraction process includes the preparation of the extraction material, separation and recovery of the solvent from extract and separation and recovery of solvent from extraction residual.

4.2. The extraction process

The extraction material is no homogeneous substance but exists of a lot of capillaries. At the beginning the solvent enters the capillaries and solutes the extract. A solution with high concentration is produced. Because of diffusion a concentration change between the solution in the extraction material and the solution surrounding the solid particles takes place. At the end of the extraction process still a certain amount of solution (consisting of solvent and extracted substance) is retained in the solid particles because of adhesive forces (= underflow). This is the reason why practically no complete extraction is possible. The solution retained in the solid

material has the same concentration on active compound as the extract (see figure 23). For equilibrium it is assumed that the whole amount of active compound is solved in the solvent.



A ... inert material
C ... active compound
B ... solvent

figure 23: ideal equilibrium

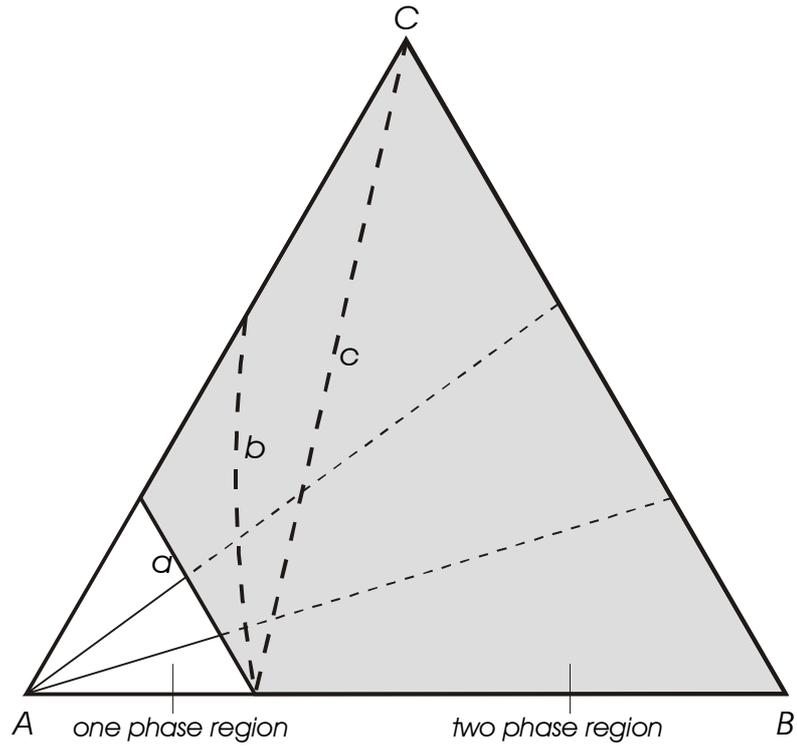
4.3. Diagrams for extraction systems

The extraction system exists of following compounds:

- pure solvent *B*
- inert material *A*: This is the whole solid material except the active substance
- active substance *C*

The equilibrium is given in the triangle diagram (see figure 24). Solvent *B* and active substance *C* are completely miscible and therefore it is a system with two mixing gaps. The connodes have to go through point *A* because extract and solution in the solid particles have the same composition so that the ratio C/B is constant.

The construction of the steps is comparable with liquid-liquid systems (see figure 25). The minimum amount of solvent is the coming closer of the mixing point to the binodal curve because all connodes cross in point *A*.



a, b, c ... binodal curves

a ... constant underflow, loading of A with B + C is constant

b ... variable underflow, loading of A changes with extract concentration

c ... constant ratio of solvent B and inert material

figure 24: equilibrium in the triangle diagram

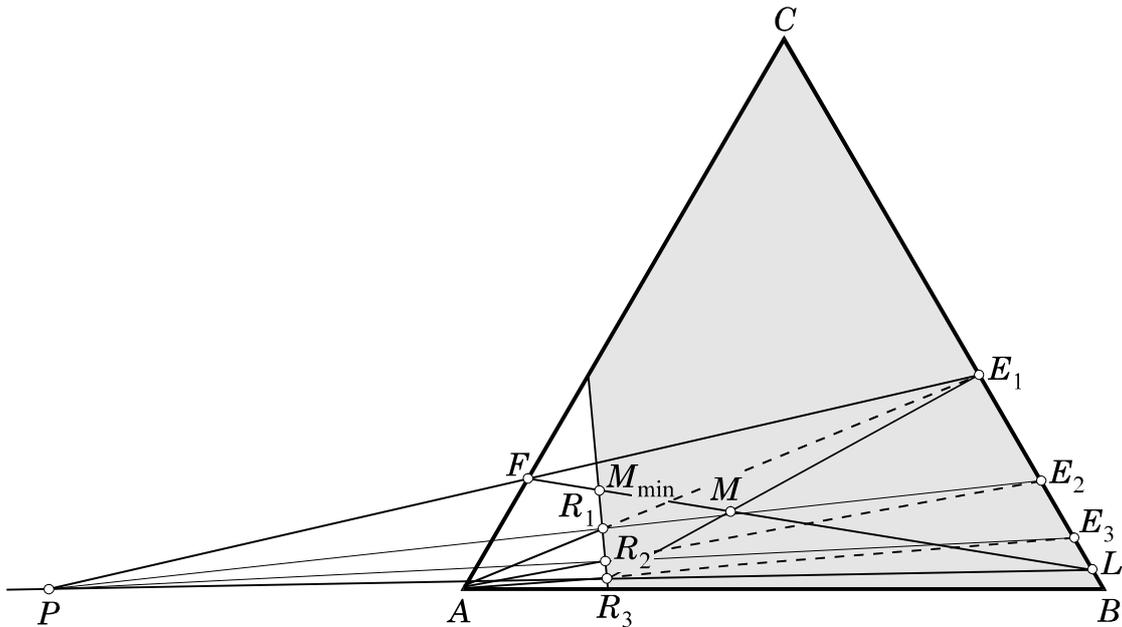


figure 25: construction of theoretical steps

4.4. Basic for high extraction rates

1. The extraction should be performed at high temperatures, because with increasing temperature normally the viscosity of the solvent and the extract decrease and on the other hand the solubility of the extract in the solvent increases.
2. The capillary ways have to be short so that only a short distance has to be overcome by diffusion. This is the reason why normally the raw material is milled for the displacement method (see chapter 4.5).

For the percolation process the solid material must be modified in that way that the solvent can easily flow through. For example oil seeds are pressed in thin flakes so that very short capillary ways are produced and further the cell walls, which include the oil material, are destroyed and a direct contact of solvent and extract is possible.

On the other hand solids in very fine form (like fish meal) have very short capillary ways but the percolation rate is very low. Therefore these fine powders are pelletised to get a granulate with good percolation properties.

3. The percolation velocity has to be high enough to wash away the extract solution which diffuses to the surface of the solid particles. By this way always a high concentration gradient between the solution in the capillaries and the solution surrounding the particle is produced. For powders the compensation of the concentration can be speeded up by mixing.
4. For multistage extractions a good efficiency of each step is achieved if the amount of miscella (= solvent B plus extracted substances C) is as low as possible. For washing machines this is achieved by centrifugation between the single washing steps. For large scale extraction plants the separation of the miscella from the underflow (= inert solid material including the miscella) is achieved by dropping zones because the effort for pressing or centrifugation between the single steps would be not economic.

4.5. Extraction processes and apparatus

4.5.1. Discontinuous extraction

For the discontinuous extraction two different methods are available:

- a. replacement process:

The extraction material is contacted with fresh solvent and extraction takes place. Afterwards the underflow is settled and the miscella is withdrawn. The underflow is contacted again with fresh solvent and so on.

disadvantage: with increasing extraction time the concentration of the miscella decreases and therefore the recycling of the solvent becomes more and more cost intensive.

application: if the extraction material does not allow another process, like sludges and powders which cannot be pelletised.

b. enrichment process:

This method is used if the raw material offers the necessary percolation properties. In most cases the solvent flows counter current to the underflow through the apparatus and by this way very high concentrations of the extract in the miscella are performed.

advantages: lower operating costs and higher through put compared to the replacement process.

4.5.1.1. Discontinuous Apparatus

1. Pot extractor (Figure 26 a)

The extractor has a volume of 2 to 10 m³ and the installed mixer is necessary to guarantee good mixing for treatment of fine materials. For structured materials the mixer is only used for evaporation of the solvent and for emptying the extractor.

2. Rotating extractor (Figure 26 b)

The extractor is filled with extraction material and solvent and starts then to rotate. The installation of heating worms and the use of a double jacket gives the possibility to evaporate the solvent at the end of the extraction cycle. By using a special form of the heating worms they can act as mixer during the extraction period.

The advantage of discontinuous extractors are the simple and robust construction of the apparatus. Disadvantages are the limited capacity and the discontinuous output of product.

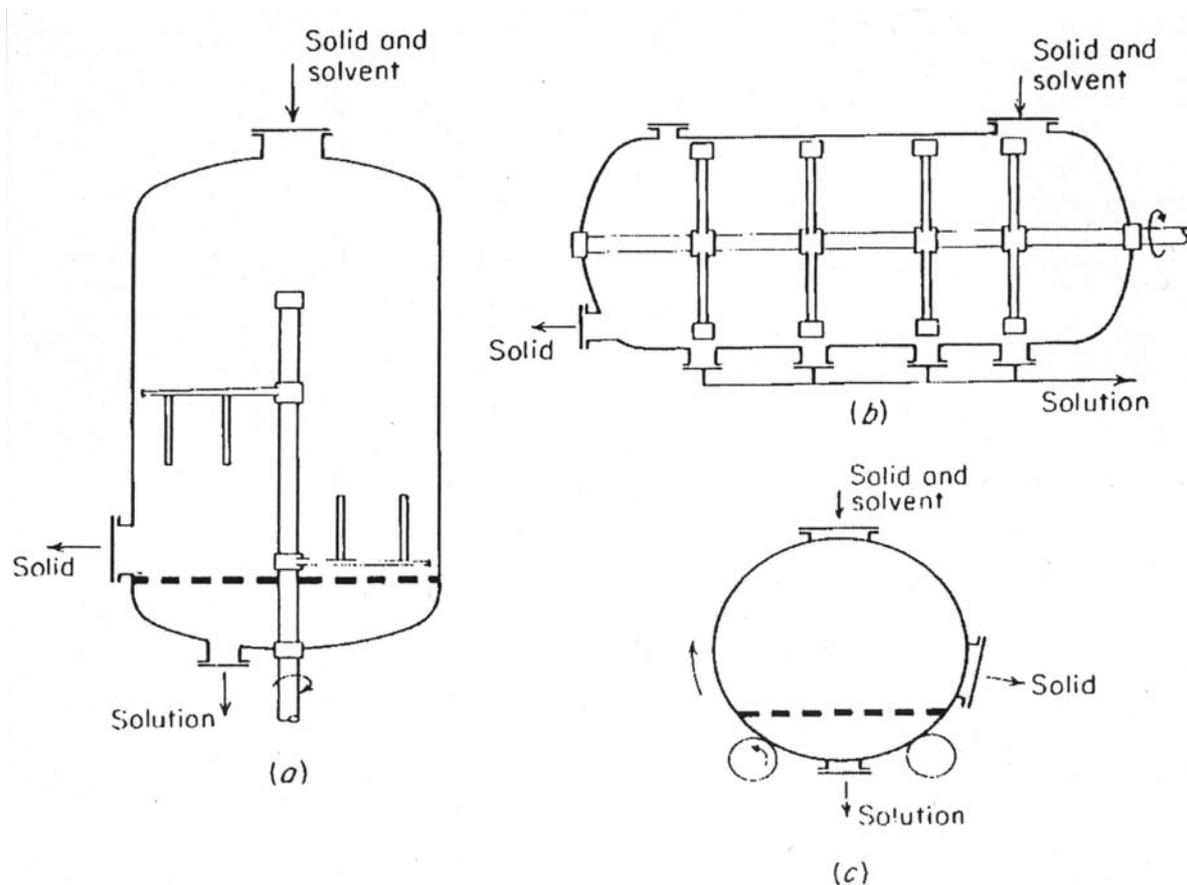


Figure 26: Pot extractor and rotating extractor

4.5.2. Continuous extraction

For the continuous operating extraction following processes are available:

1. Percolating Process:

The solvent passes through the non moving solid material and extracts the soluble substances. The basic for this process is that the material has good percolating properties, which means that the solvent can pass easily the solid material. The advantage of this method is that the mechanical treatment of the solid material is low because of no movement and further during the passing through of the solution a self filtration takes places resulting in a minimum content of fine solid particles in the extract solution.

2. Immersion method:

For this process the solid materials dips completely into the solvent and is mixed with this. Therefore no special percolation properties of the solid material are necessary. The disadvantage of this method is that no self filtration of the extract solution takes place and therefore a filtration step has to be installed before the distillation step.

Generally it is the aim to combine the advantages of both methods.

The advantages of the continuous operating extraction process are

- large amount of solid material can be treated in apparatus of compact size
- even at low residual content of active agents in the residual material extract solutions with high active agent concentrations are produced with a low amount of solvent
- short extraction times because no dead times arise as for the discontinuous process
- low content of fine solid particles in the extract solution so that this solution has not be filtrated before further treatment
- an optimal heat balance is achieved if for evaporation of the solvent the heat amount of the exhaust vapour is used

4.5.2.1. Continuous extraction apparatus

1. Continuous horizontal extractor: (Figure 27)

The solid material is given into baskets and is contacted by the percolation method with solvent. The flow of the solvent through the extractor is counter current to the solid material flow.

2. Hildebrandt extractor: (Figure 28)

The solid material is extracted according to the immersion method. screw conveyors are installed in the extractor for transporting the solid material. Again the solvent flows counter current to the solid materials through the extractor.

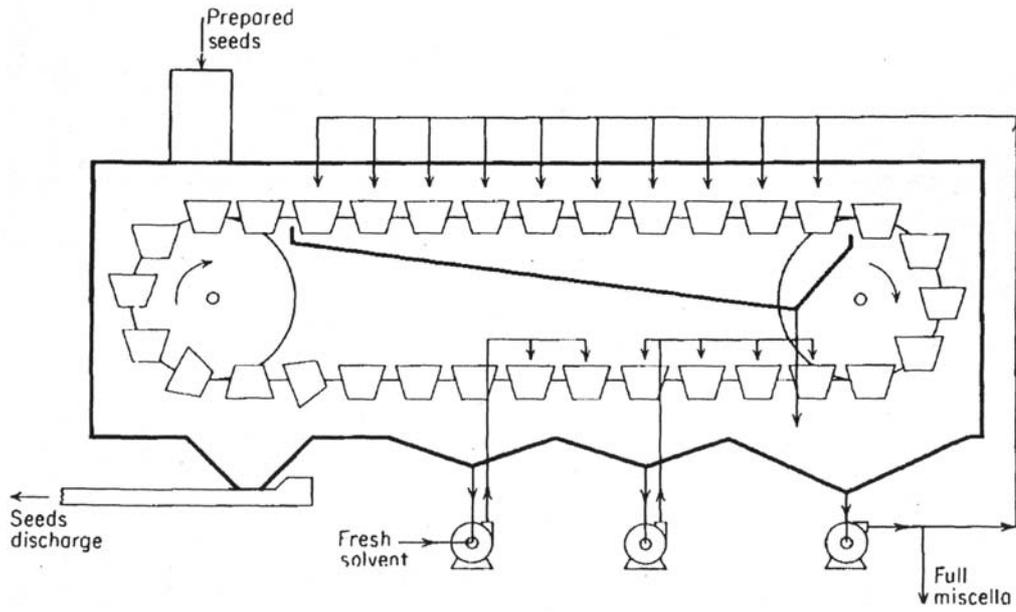


Figure 27: Continuous horizontal extractor

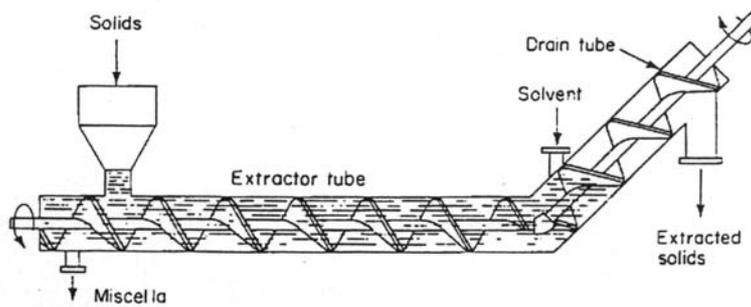
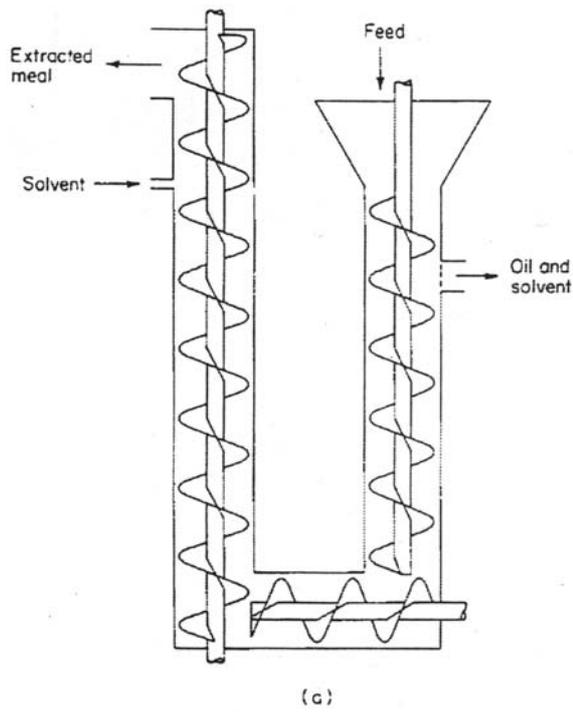


Figure 28: Hildebrandt extractor

3. Bonotto extractor: (Figure 29)

Is used for counter current extraction following the immersion method. The solid material is transported by the mixer on the tray until it reaches the open sector where the solid material falls down on the next tray. The screw conveyor at the outlet withdraws the extracted solid material (underflow) and prevents the flowing out of the solution from the extractor.

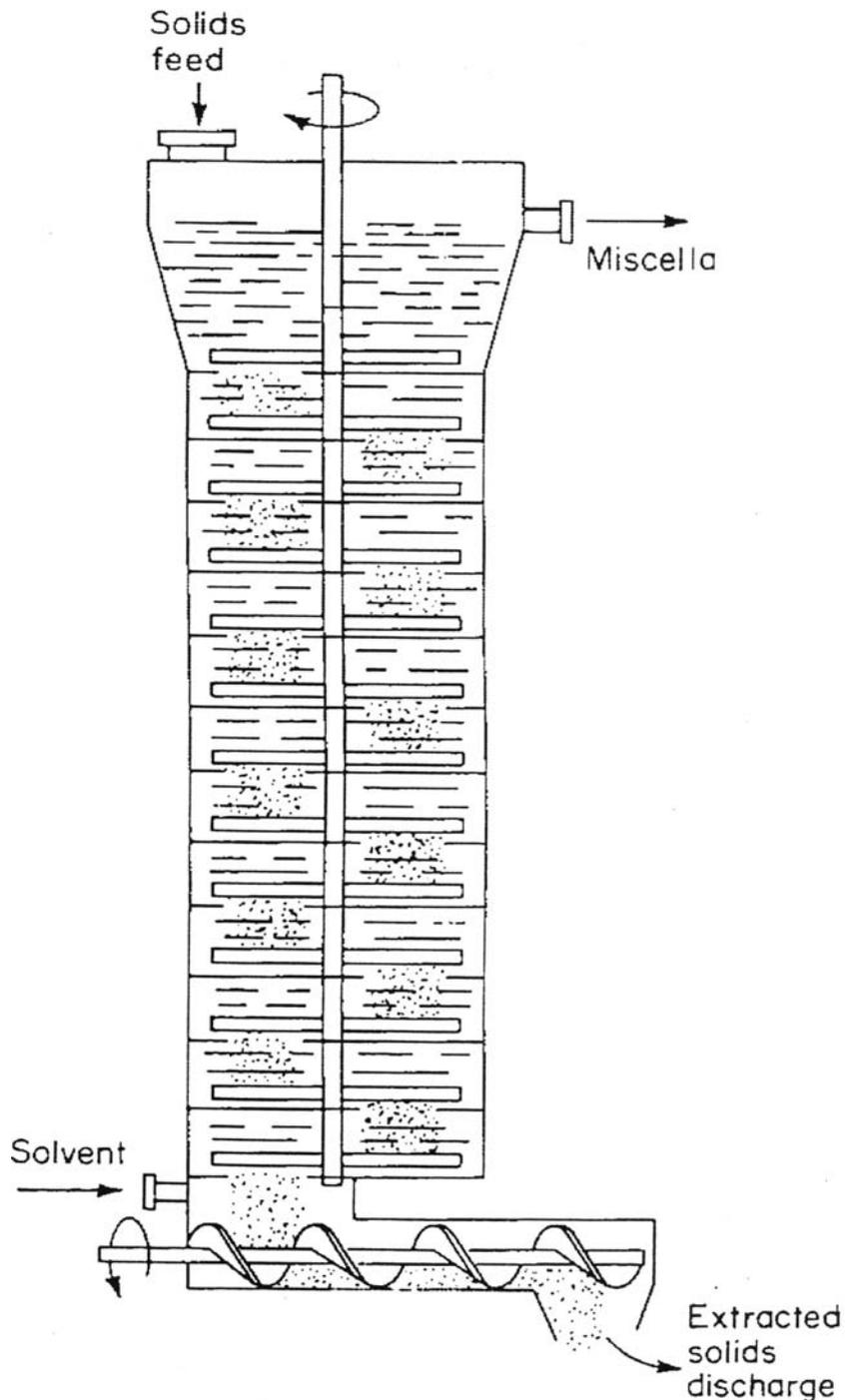


Figure 29: Bonotto extractor

4. Bollmann extractor: (Figure 30)

The fresh solvent is added during the upward movement of the baskets so that this part operates counter current. The already preloaded solution is withdrawn from the bottom of the extractor and enters the downward moving baskets so that this part of the extractor operates in a cocurrent way. The full miscella is withdrawn at the bottom of the extractor. In the baskets self filtration takes place, so that no further treatment of the miscella before distillation is necessary.

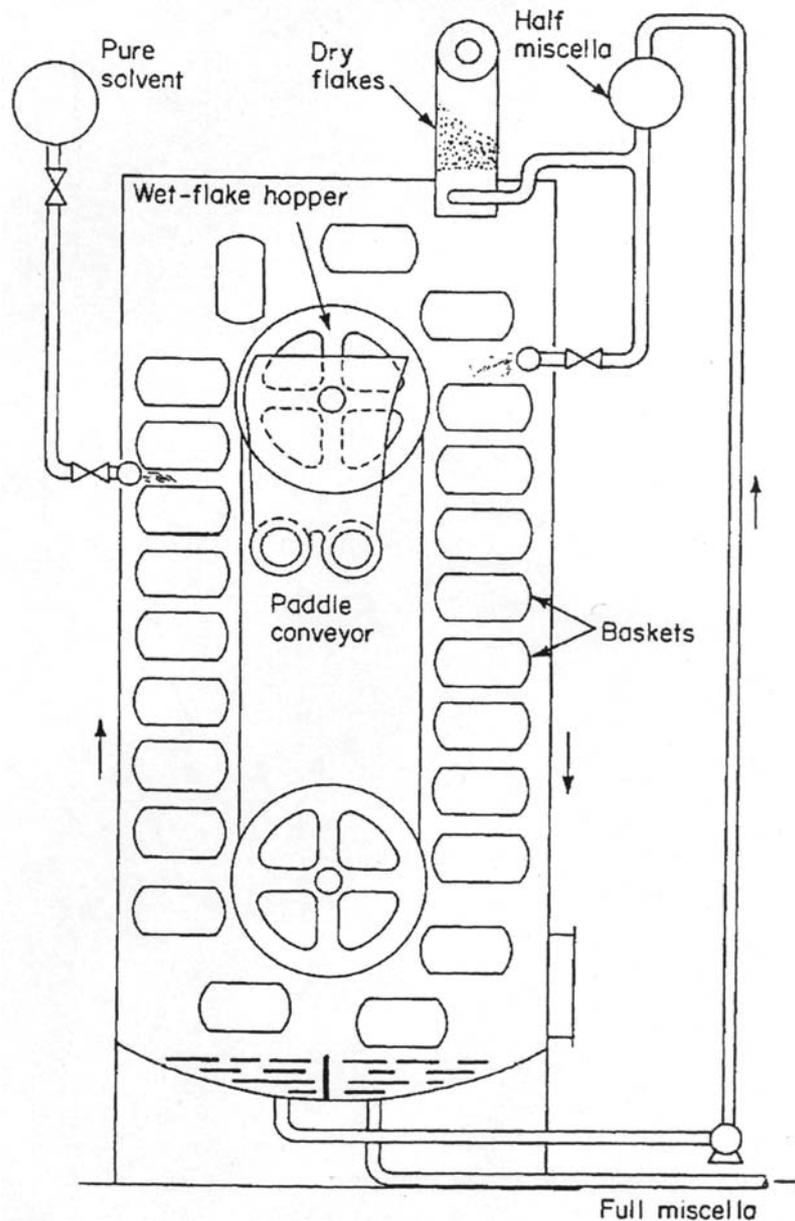


Figure 30: Bollmann extractor

5. Kennedy extractor: (Figure 31)

The solid material is transported by paddles from one chamber to the next and this in counter current way to the solvent. The chamber where the miscella is withdrawn is used as filtration step, where fine particles are separated from the extract solution.

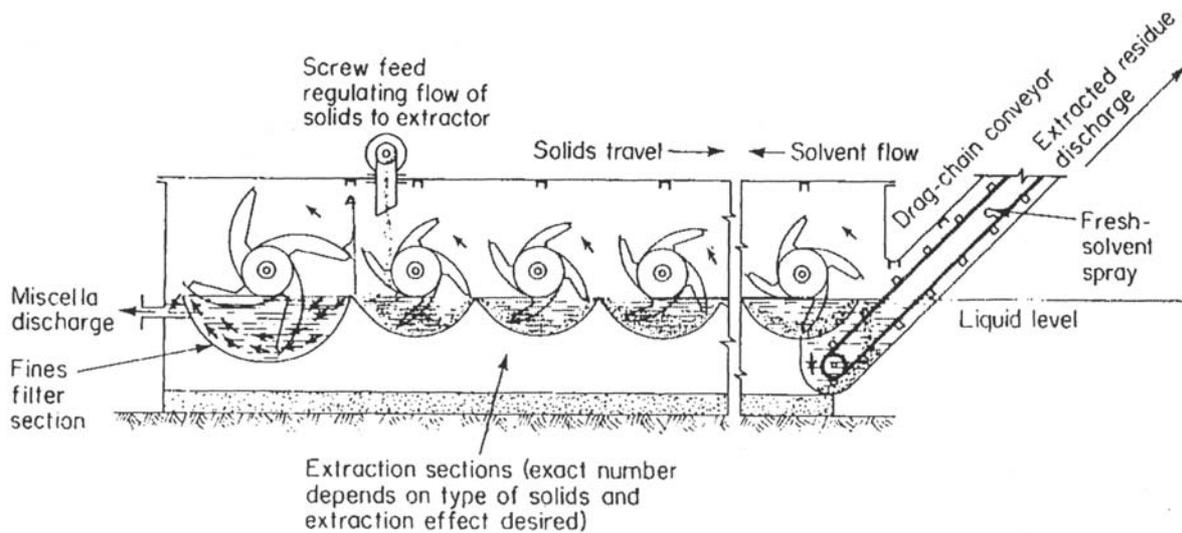


Figure 31: Kennedy extractor

6. Rotocell- or Carousel extractor: (Figure 32)

This extractor exists of a fixed tray and of a rotating star wheel, which produces the single extraction chambers. The solid material is filled in the first chamber and contacted with preloaded solvent according the percolation method. At given time interval the solvent input is stopped, during a certain time interval the miscella can drop down from the solid material, then the rotating star wheel turns to next position and the solvent input is started again. The counter current flow of the solvent is achieved by pumping the solvent of one step to the next contrary to the solid flow. For the removal of the solid material in one segment no tray is installed so that the solid falls down and is withdrawn.

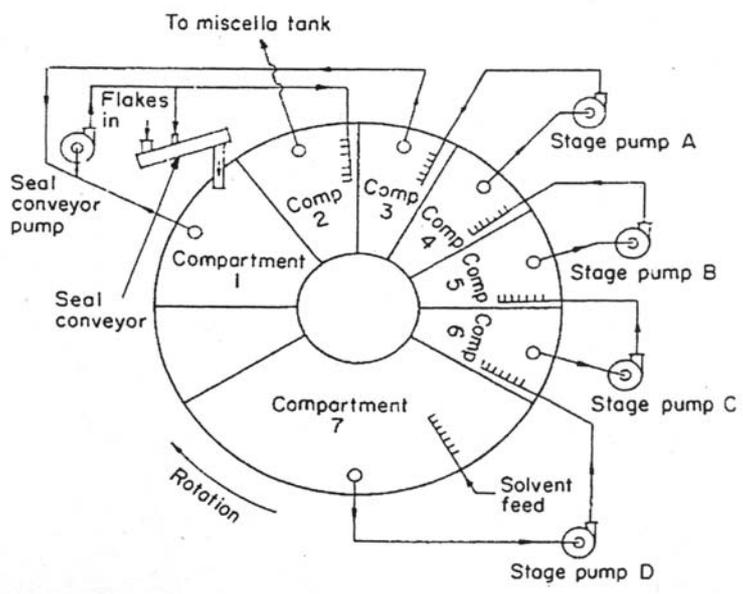
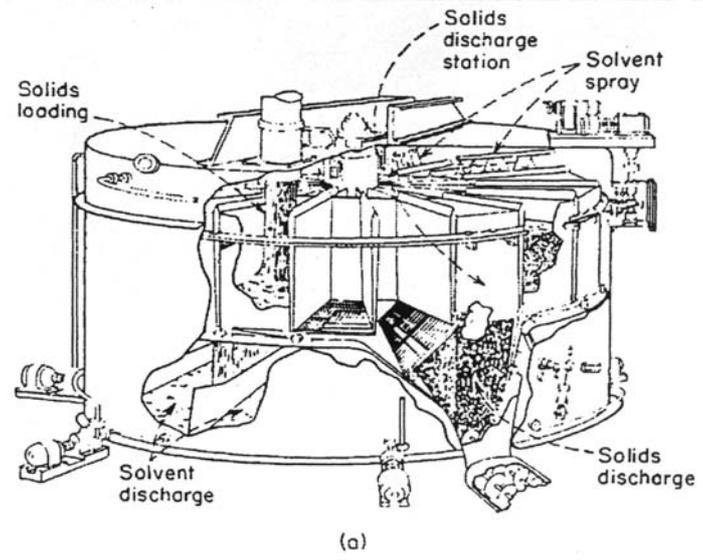
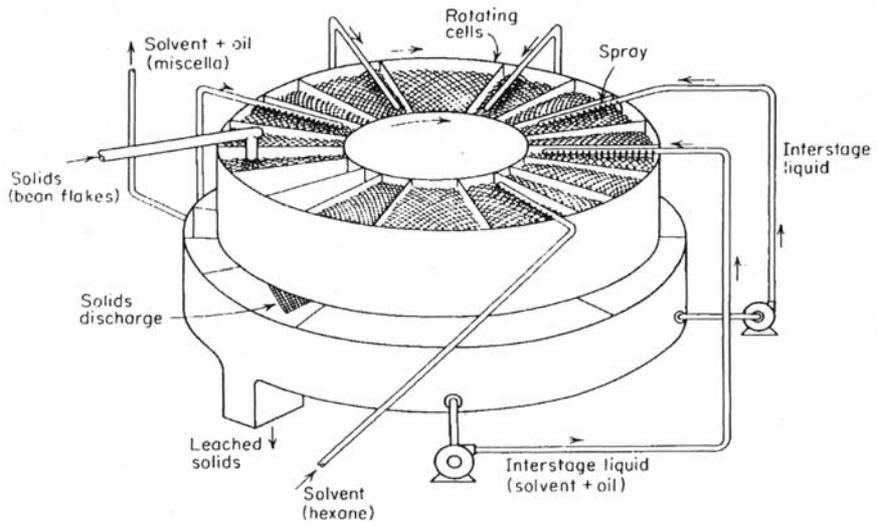


Figure 32: Rotocel or Carousel extractor

7. Lurgi extractor: (Figure 33)

This extractor exists of a frame belt which lays in the upper and lower section of the extractor on a sieve belt. The sieve belts are the bottom of the extraction chambers. The upper sieve belt is shorter than the frame belt so at this section no bottom for the extraction chamber exists and the solid material falls down in the next chambers. The upper and lower part of the extractor are separated so that the miscella can be collected separately and transported to the step before, realising the counter current flow.

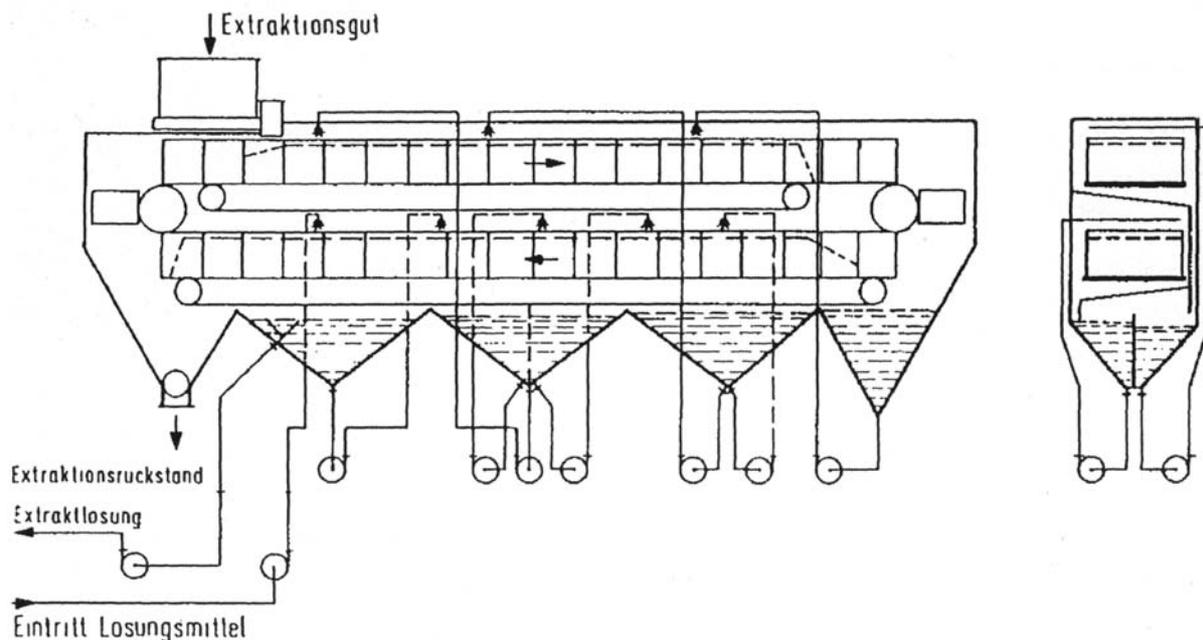


Figure 33: Lurgi extractor

8. DeSmet extractor: (Figure 34)

The input of the solid material is done by a pressure lock and the solid material lays on a sieve belt. The height of the solid layer is regulated by a sliding piece. After the dropping zone the underflow is withdrawn by a lock. Because of the closed system it is the advantage of the DeSmet extractor that it can be operated at higher pressure and therefore at higher temperatures.

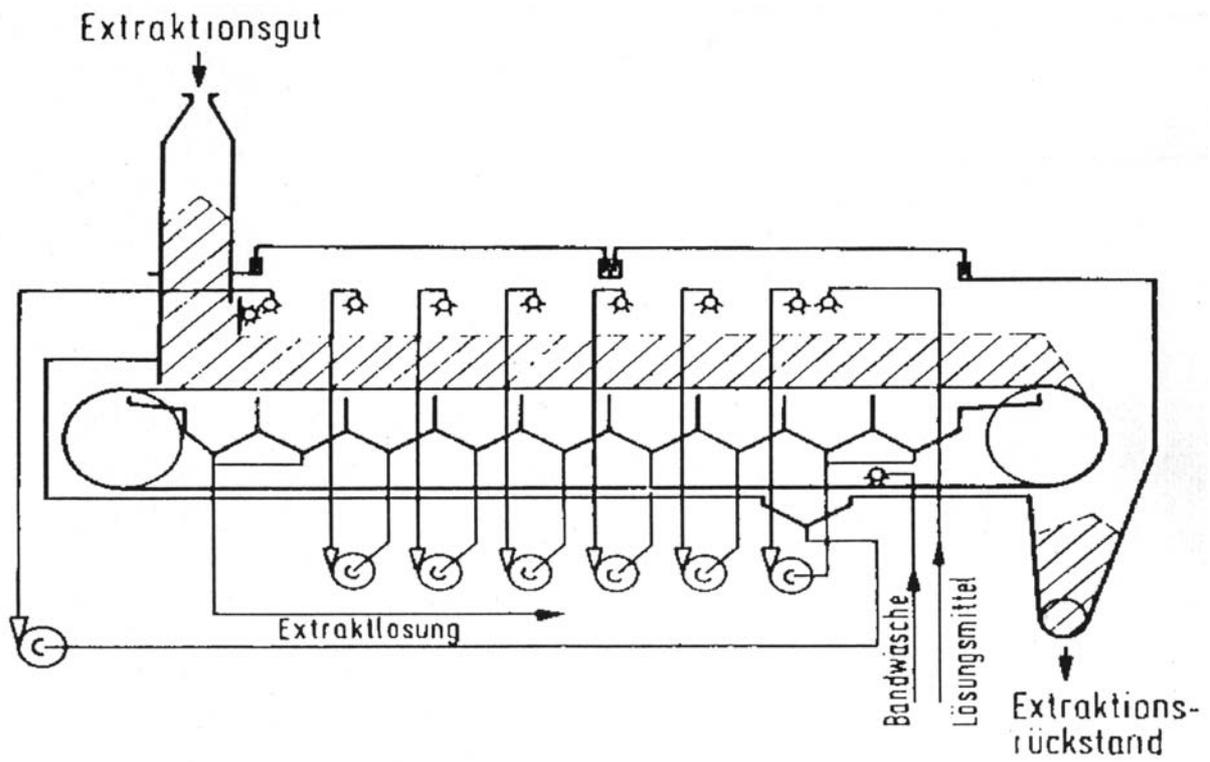


Figure 34: DeSmet extractor

5. High pressure extraction (HPE)

For liquid-liquid and solid-liquid extractions normally organic solvents are used. For the high pressure extraction a densified gas is used as solvent. The pressure of the gas has to be always higher than the critical pressure but for the temperature as well sub- as supercritical conditions are used. If both parameters are supercritical a fluid is present and if temperature is subcritical a liquid is present. Table 1 give a comparison of physically properties of a liquid, gas and fluid.

table 1: comparison of physically properties

	gas	fluid	liquid
density [kg/l]	10^{-3}	0,3 – 0,9	1
diffusion coefficient [cm^2/s]	10^{-1}	10^{-3} – 10^{-4}	10^{-5}
viscosity [g/cm s]	10^{-4}	10^{-4} – 10^{-3}	10^{-2}

The density of a supercritical fluid is comparable to the density of liquids but the viscosity of the fluid is like a gas and the diffusion coefficient is one to two orders higher than of a liquid. Therefore supercritical fluids are able to penetrate into a solid, solve substances and transport them from the inner part to the surface.

A further advantage of supercritical fluids is that heavy volatile substances show a high solubility in supercritical fluids and the solubility can be influenced by variation of pressure and temperature, which have different influences on the solubility. An increase in pressure results always in higher solubility because of the increase in density. An increase in temperature has two influences: on one side the density of the fluid decreases and on the other side the vapour pressure of the substance to be solved increases. Depending which effect is dominant an increase in temperature can result as well in an increase as in a decrease of solubility. Normally for pressures higher twice the critical pressure an increase in temperature results also in an increase in solubility.

The gas which is most used for high pressure extraction processes is carbon dioxide (CO_2) because it is inflammable, non toxic and available in large amounts with a low price. In this apolar solvent organic oxygen compounds with low molecular weight (ketones, esters, alcohols, aldehydes) have a high solubility, most of the non polar organic compounds (alkanes, alkenes, terpenes) have a good solubility and polar organic substances with high molecular weight are difficult to solve.

Depending on the state of aggregation of the raw material different extraction apparatus have to be used. Up to now no continuous in- and output of solid materials for extraction apparatus are available because of the high pressure and therefore extraction of solid materials is operated discontinuously. For a quasi continuous flow of the solids more extractors in cascade operation are used. For liquid raw materials high pressure extraction columns are used which are operated in counter current flow.

For separating the extracted substances from the extraction fluid different possibilities exist. Expanding the fluid (see figure 35) results in density reduction and therefore reduction of the solvent power. Therefore the substances solved in the fluid are separated in the separator.

A further method is changing of temperature for separation of substances from the extraction fluid. Depending on the solubility behaviour the fluid leaving the extractor has to be heated or cooled depending at which temperature solubility decreases (see figure 36). After the separator the extraction fluid is brought again to extraction temperature. The advantage of this method is an isobarically process and the disadvantage is that no total separation can be achieved and therefore preloaded fluid enters the extractor.

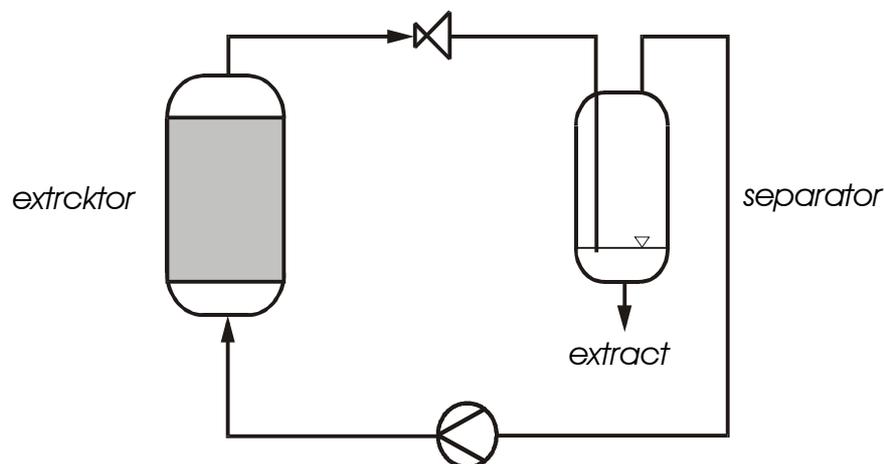


figure 35: separation by pressure reduction

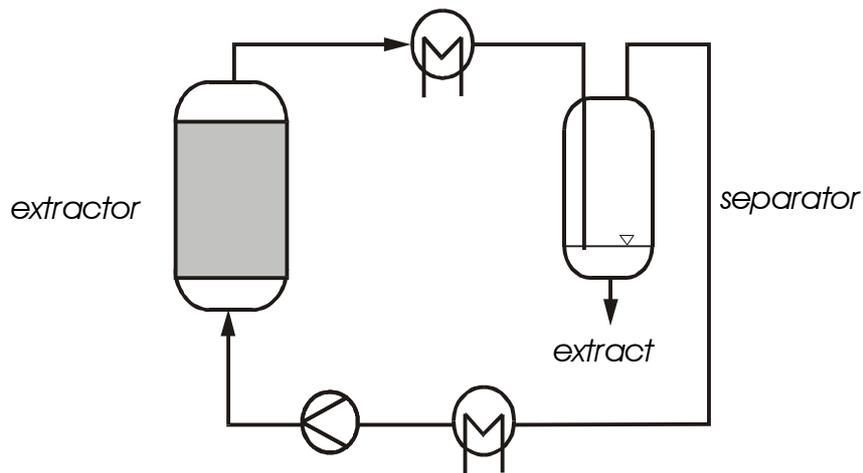


figure 36: separation by temperature change

In most cases a combination of pressure and temperature change is used. The extraction fluid is depressurised in the two phase region and afterwards heated up to gaseous state. At this condition the fluid has no solvent power and complete separation is achieved. Afterwards the fluid is condensed and compressed again so that the solvent circle is closed.