

Liquid-Liquid Extractor Design

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Introduction

Liquid extraction (or solvent extraction) refers to an operation in which the components of a liquid mixture are separated by contacting it with a suitable insoluble liquid solvent which preferentially dissolves one or more components. In this operation, the separation of the components depends upon the unequal distribution of the components between the immiscible liquids. The feed solution represents one phase and the solvent to be used to effect separation represents the second phase. The mass transfer of the solute liquid takes place from the feed solution to the solvent phase.

Both the distillation and liquid-liquid extraction are used for the separation of the constituents of a liquid mixture. In order to achieve a separation by distillation, as well as by liquid-liquid extraction, it is necessary to have two phases. In distillation it is necessary to have liquid and vapor phases and heat is used to produce the vapor. In liquid-liquid extraction it is necessary to have two liquid phases and the solvent is used to produce another liquid phase. The solvent used in liquid-liquid extraction is analogous to the heat used in the distillation.

Distillation and extraction both are used for the separation of the constituents of any liquid mixture based on the economics evaluation of the individual methods.

Table 1: Comparing Extraction and Distillation

Extraction	Distillation
1. Extraction is an operation in which constituents of the liquid mixture are separated by using an insoluble liquid solvent	1. Constituents of the liquid mixture are separated by using thermal energy



2. Extraction utilizes the differences in solubilities of the components to effect separation	2. Utilizes the differences in vapor pressures of the components to effect separation
3. Selectivity is used as a measure of degree of separation	3. Relative volatility is used as a measure of degree of separation
4. A new insoluble liquid phase is created by addition of solvent to the original mixture	4. A new phase is created by addition of heat
5. Phases are hard to mix and harder to separate	5. Mixing and separation of phases is easy and rapid
6. Extraction does not give pure product and needs further processing	6. Gives almost pure products
7. Offers more flexibility in choice of operating conditions	7. Less flexibility in choice of operating conditions
8. Requires mechanical energy for mixing and separation	8. Requires thermal energy
9. Does not need heating and cooling provisions	9. Requires heating and cooling provisions
10. Often a secondary choice for separation of components of liquid mixture	10. Usually the primary choice for separation of components of liquid mixture

Whenever separation by both distillation and extraction is possible the choice is usually distillation, irrespective of heating and cooling requirements. When extraction is used, the solvent should be recovered for reuse and hence extraction is usually followed by distillation for the recovery of solvent. The combined operation is more complicated and more expensive than ordinary distillation. However, when the separation of the components via distillation is difficult, extraction can be an attractive alternative.

Typical liquid-liquid extraction operations utilize the differences in the solubilities of the components of a liquid mixture. The basic steps involved include:

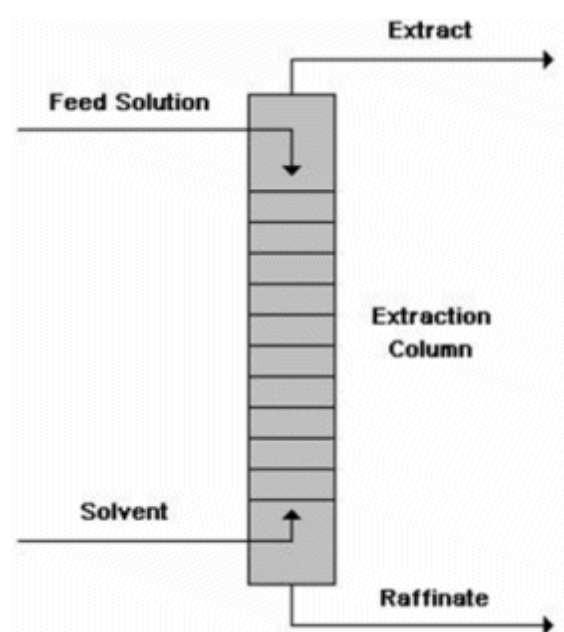
1. Contacting the feed with the extraction solvent.
2. Separation of the resulting phases, and
3. Removal/recovery of solvent from each phase.

The liquid mixture to be treated and a suitable, insoluble solvent are contacted intimately.

The constituents of liquid mixtures are distributed between the two phases resulting in some degree of separation (which can be improved by a multistage contact) and the phases are separated from one another based on the density differences of the liquid phases. For example, acetone may be preferentially extracted from a solution in water with the help of chloroform. The resulting chloroform phase contains a large part of acetone, but little water.

The solution to be extracted is called the feed. The liquid extraction liquid is called the solvent. The residual liquid solution from which the solute is removed is called the raffinate. The extracted solvent rich product is called the extract. The extract phase contains the desired product in a larger proportion.

Thus, if a solution of acetic acid in water is contacted with a solvent such as ethyl acetate then two phases will result. The extract (ester/organic layer) will contain most of acetic acid in ethyl acetate with a small amount water. The raffinate (aqueous layer) will contain a weak acetic acid solution with a small amount of ethyl acetate. The amount of water in the extract and ethyl acetate in



the raffinate depends upon their solubilities in one another.

The Distribution Coefficient

In dilute solutions at equilibrium, the concentration of the solute in the two phases is called the distribution coefficient or distribution constant 'K'.

$$K = C_E / C_R$$

Where the C_E and C_R are the concentrations of the solute in the extract and the raffinate phases respectively. The distribution coefficient can also be given as the weight fraction of the solute in the two phases in equilibrium contact:

$$K' = y^* / x$$

Where y^* is the weight fraction of the solute in the extract and x is the weight fraction of the solute in the raffinate. In liquid-liquid extraction, the liquid-liquid equilibrium must be considered. This is best represented by equating the chemical potential of both liquid phases:

$$\mu_i^{II} = \mu_i^{III}$$

This relationship reduces to an expression, which is dependent only on the liquid mole fractions and their respective activity coefficients:

$$\gamma_i^{II} x_i^{II} = \gamma_i^{III} x_i^{III}$$

Activity coefficient models, such as UNIFAC, UNIQUAC (universal quasichemical), and NRTL can be used to determine the mole fractions. All three models above are applicable to liquid-liquid equilibrium, the choice of which model to use depends on which properties are available. For a multi-component system, the UNIQUAC equation for the liquid-phase activity coefficient is:

$$\ln \gamma_i = \ln \gamma_i(\text{combinatorial}) + \ln \gamma_i(\text{residual})$$

The combinatorial and residual activities are based on the statistical mechanical theory which describes how local compositions result from the size and energy differences between the molecules in the mixture.

When to Chose Liquid-Liquid Extraction

- When large volumes of water must be removed to complete a separation. The large latent heat of vaporization of water can make this an energy intensive process.
- When two or more liquids form a close boiling azeotrope, the desired final concentration may not be possible via distillation.
- When one or more of the components are considered thermally sensitive or unstable, distillation may not be a feasible option.

In such cases, extraction is attractive and often preferred. The separation of acetic acid from dilute solutions of water is usually more economical via extraction followed by distillation. Distillation would be feasible for this separation, but by using extraction first, the amount of water that must be vaporized is reduced significantly.

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Rohit Ramesh Rewagad and Piyush Khataavkar
Bachelor of Technology in Chemical Eng.
Laxminarayan Institute of Technology, Nagpur India
Email: piyushkhataavkar "at" gmail.com



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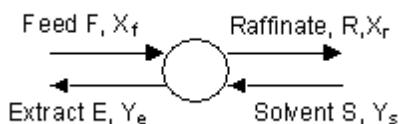
Modes of Operation: Cross-Current

Cross-current mode is mostly used in batch operation. Batch extractors have traditionally been used in low capacity, multi-product plants typically found in the pharmaceutical and agrochemical industries. For washing and neutralization operations that require very few stages, cross-current operation is particularly practical and economical and offers a great deal of flexibility. The extraction equipment usually includes an agitated tank that may also be used for the reaction steps. In these tanks, the solvent is first added to the feed, the contents are mixed, settled, and then separated. Single stage extraction is used when the extraction is fairly simple and can be achieved without a high amount of solvent. If more than one stage is required, multiple solvent-washes will be required.

Though operation in cross-current mode a great deal of flexibility, it must be examined carefully due to the high solvent volume requirements and low extraction yields. The following illustration gives a quick method to calculate solvent requirements for cross-current extraction.



A single-stage extractor can be represented as:



F = Feed quantity / rate, mass

R = Raffinate quantity / rate, mass

S = Solvent quantity / rate, mass

E = Extract quantity / rate, mass

X_f , X_r , Y_s , and Y_e are the weight fractions of solute in the feed, raffinate, solvent and extract, respectively.

Partition coefficient 'm' is defined as the ratio of Y_e to X_r at equilibrium conditions

The flows and concentrations are represented in solute-free basis as such a representation leads to simplification of equations.

For example, for a 100 kg/hr feed containing 10% weight acetic acid, $F = 100 - 10 = 90$ kg/hr, $X_f = 0.1 / (1 - 0.1) = 0.111$

The component mass balance can be represented as:

$$F X_f + S Y_s = R X_r + E Y_e$$

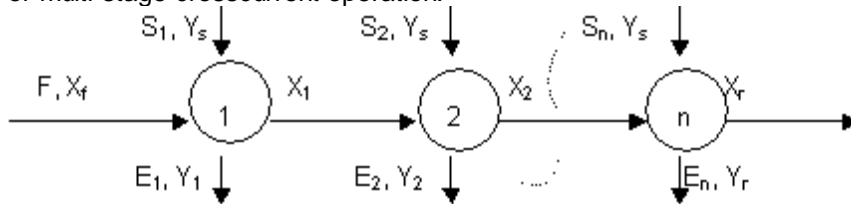
Assuming (i) immiscibility of feed and solvent and (ii) the initial solvent is free of solute, i.e., $F = R$, $S = E$ and $Y_s = 0$ and using the equilibrium relation of $Y_e = m X_r$, this equation simplifies to

$$S = F/m (X_f / X_r - 1)$$

Or

$$\text{Reduction ratio, } X_f / X_r = 1 + m S/F$$

For multi-stage crosscurrent operation:



Assuming that the partition coefficient (m) is constant over the concentration range and the solvent quantity in each of the 'n' stages is the same, i.e., $S_1 = S_2 = \dots = S_n = S/n$,

Solvent Requirement is

$$S = n * F/m [(X_f / X_r)^{1/n} - 1]$$

Or

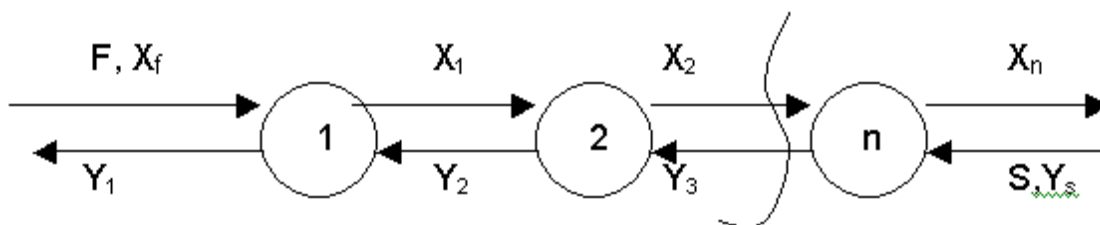
$$\text{Reduction ratio } X_f / X_r = (1 + mS/nF)^n$$

It can be proved mathematically that the total solvent quantity would be minimum if the solvent were distributed equally between washes.

Reference: Optimize Liquid-Liquid Extraction, R. Madhavan, Cheresources.com

Modes of Operation: Counter-Current

As described above, the cross-current operation is mostly used in low capacity, multi-product, batch plants. For larger volume operations, a counter-current configuration is often used. Counter-current patterns are capable of more efficient use of the solvent. Counter-current mixer-settlers or columns are usually employed. Countercurrent operation conserves the mass transfer driving force thus they typically yield optimal performance.



Equations for countercurrent extraction get more complicated with increasing number of stages. It can be shown that for a 'n' stage operation, the raffinate concentration would be

$$X_r = X_f * (mS/F - 1) / ([mS/F]^{n+1} - 1)$$

The solvent requirement for any raffinate concentration X_r could be determined by iteration from the above equation.

For $mS/F = 1$, the equation takes the form of $X_r = X_f / (n + 1)$

Reference: *Optimize Liquid-Liquid Extraction*, R. Madhaven, Cheresources.com

The dimensionless term mS/F , included in all the above equations, is called the extraction factor (E), and is an important parameter in the design of extraction processes. For a given number of stages, the higher the E factor, the higher the reduction ratio thus a better extraction. Systems with E of less than 1.3 are not likely to be commercially feasible.

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Rohit Ramesh Rewagad and Piyush Khatavkar
Bachelor of Technology in Chemical Eng.
Laxminarayan Institute of Technology, Nagpur India
Email: piyushkhatavkar "at" gmail.com





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Operational Conditions

The following are typically key operational parameters for an extraction process:

Operating Temperature

Operating Pressure

Residence Time

Feed Flow Rate

Composition

Temperature of entering stream

Pressure of entering stream

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As in many separation processes, the pressure and temperature play a large role in the effectiveness of the separation. In order for a good split of the feed stream, vaporization must be prohibited.

The process will be adversely affected if one or more of the components are allowed to vaporize. In addition, the temperature should be high enough that the components are all soluble with one another. Moderate temperatures can facilitate the selection of a suitable solvent. With these rules in mind, most extraction processes can proceed at or near ambient temperature and pressure which is a key advantage of extraction.

Temperature can also be used as a variable to alter selectivity. Elevated temperatures are sometimes used to keep viscosity low minimizing mass-transfer resistances. Other parameters to be considered are selectivity, mutual solubility, precipitation of solids, and vapor pressure.

In many applications, a "gently" separation process is needed as an high temperatures could destroy the desired product (pharmaceutical industry). For these applications, extraction is ideal since the only temperature requirement is that dictated by the solubility. The challenge in these cases is to find a suitable solvent for the extraction.

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Operating pressure has a negligible effect on extraction performance and therefore most extractions take place at atmospheric pressure unless governed by vapor pressure considerations.

Residence time is an important parameter in reactive extraction processes (e.g., metals separations, formaldehyde extraction from aqueous streams) and in processes involving sensitive components (e.g., antibiotics & vitamins).

Selecting a Solvent

Any liquid to be used as a solvent will not possess all the properties considered desirable for extraction and hence compromise is usually necessary for selecting the best solvent. While selecting a solvent for extraction, qualities such as selectivity, recoverability, distribution coefficients, and density should be given careful consideration.

- Selectivity – The ratio of the concentration of solute to feed solvent in the extract phase to that in raffinate phase is called the selectivity or separation factor. It is a measure of the effectiveness of the extraction solvent for separating the constituents of feed. Selectivity should be greater than 1 for all extraction operations. If it is equal to 1, then separation by extraction is not possible.
- Recoverability - As the solvent must be recovered for reuse (usually by distillation), the formation of an azeotrope with the extracted solute is not desirable. To minimize recovery costs, the relative volatility should be high. The latent heat of vaporization of the solvent should be as small as possible whenever the solvent is to be vaporized.
- Density – A difference in the densities of saturated liquid phases should be as large as possible for physical separation of the phases.
- Interfacial Tension – The interfacial tension should be higher for the coalescence of droplets to occur more readily.
- The solvent should be inexpensive, non-toxic, and non-flammable if possible. The solvent should have a low viscosity, freezing point, and vapor pressure, for ease in handling and storage. The solvent should be chemically stable and inert to the other components.

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Rohit Ramesh Rewagad and Piyush Khataavkar
Bachelor of Technology in Chemical Eng.
Laxminarayan Institute of Technology, Nagpur India
Email: piyushkhataavkar "at" gmail.com



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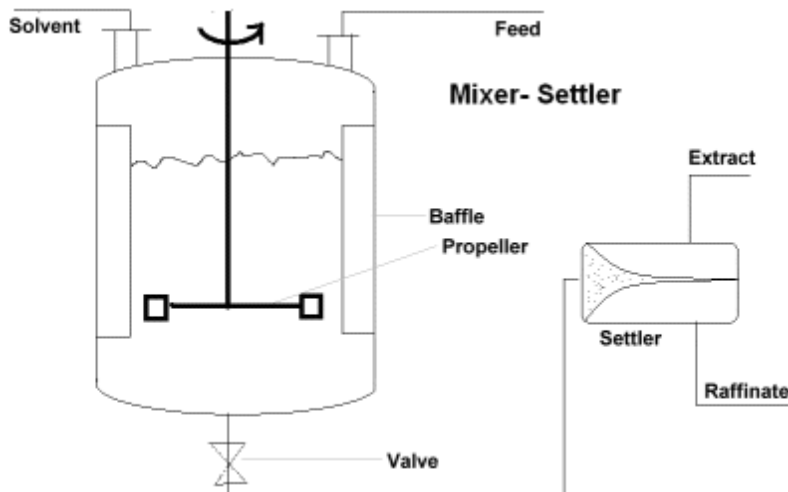
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Extraction Equipment

Industrial extraction equipments can be classified as stage type extractors or differential/continuous contact extractors. In stage type extractors, the two phases are allowed to mix together so as to reach equilibrium, then they are incrementally separated while being passed counter-current to one other. The advantages of stage type contactors include simplicity of design, no axial mixing, and high stage efficiency. These units are large and bulky due to separation performed after each stage. In differential extractors, the two phases are always in continuous contact. Differential extractors are compact and required minimal floor space.

Mixer-Settler

Mixer-settlers may be used in batch mode (a single unit) or in continuous mode by staging multiple units. A mixer-settler consists of a vertical tank incorporating a turbine or a propeller agitator. Charging nozzles are located at the top and discharge connection



with a sight glass at the bottom. The feed solution to be extracted is charged into the vessel and the necessary amount of solvent is added. The vessel is agitated for a predetermined amount of time and at the end of mixing cycle, agitation is stopped and the phases are allowed to settle. Finally, the raffinate and extract phases are withdrawn from a separate receiver vessel.

The main disadvantage of the mixer-settler is that it requires a large vessel with high liquid volume demands. Advantages include the high stage efficiency, flexibility, ability to handle high viscosity liquids, and its high capacity.

Contacting Columns

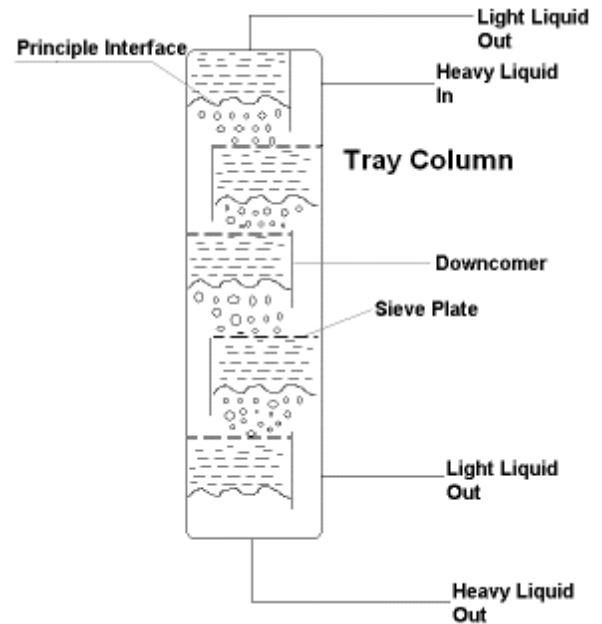
Contacting columns are practical for most liquid-liquid extraction systems. The packing, trays, or sprays typically used increase the surface area in which the two liquid phases contact. This also allows for a longer flow path. In the selection of packing, it is necessary to select a material that is wetted by the continuous phase. In column extractors, the phase with the lower viscosity (lower flow resistance) is generally chosen as the continuous phase. Also note that the phase with the higher flow rate can be dispersed to create more interfacial area and turbulence. This is accomplished by selecting an appropriate material of construction with the desired wetting characteristics. In general, aqueous phases wet metal surfaces and organic phases wet non-metallic surfaces. Changes in flows and physical properties along the length of extractor should also be considered.

Tray Columns

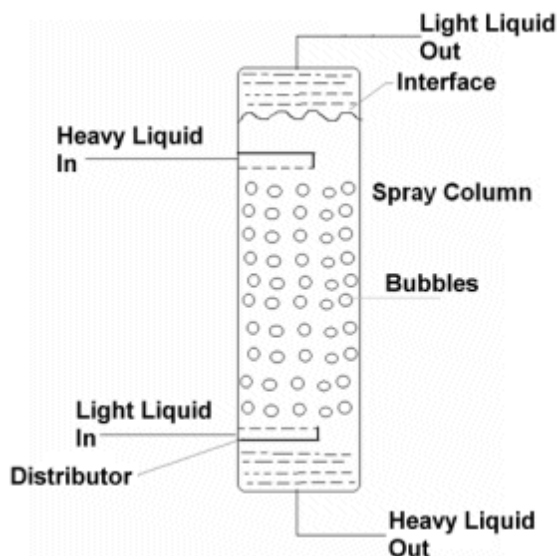
Tray columns are multi-staged, counter-current contactors where the axial mixing of the continuous phase is confined to the region between trays, and dispersion occurs at each tray resulting into effective mass transfer. Tray columns are very effective, especially for systems of low-interfacial tension, with respect to their liquid handling capacity and extraction efficiency. The perforations in the plates are about 1.5 to 4.5 mm in diameter and the plate/tray spacings are usually 150-600 mm.

Generally the arrangement of plates and down comers is the same as that used for gas-liquid contact, except that no weir is needed. The light liquid is introduced at the bottom, passes through the perforation in the plate in the form of the fine droplets. The fine droplets rise through the heavy continuous phase (being fed into the top) and coalesce into a layer beneath the plate and then are again dispersed through the plate above.

Heavy continuous phase liquid is introduced at the top, passes across the each plate and flows downward from a plate above to the plate below via downcomer. Principle interface is maintained at the top, light liquid is removed from the top and the heavy liquid from the bottom.



Spray Columns



Spray columns consists of an empty tower with inlet and outlet connection at the top and bottom, for introducing and removing the heavy and light liquid phases. Since the spray column is essentially an empty vessel, there will be axial mixing making it difficult to obtain the equivalent of more than one or two theoretical stages.

Spray towers are operated in one of two distinct ways: either the light phase or the heavy phase may be dispersed. When the light phase is dispersed, it is introduced through the bottom nozzle, droplets rise through the heavy phase and finally coalesce to form a liquid-liquid interface at the top. When the heavy phase is dispersed, it is introduced from the top, and the principle interface will be at the bottom.

Advantages of the spray column include construction simplicity and a high throughput per unit area. Disadvantages include its low efficiency due to axial mixing in the continuous phase and the need for a tall tower to achieve the degree of separation needed. Spray tower performance is generally considered poor because of its considerable recirculation of the continuous phase with little turbulence.

Packed Towers

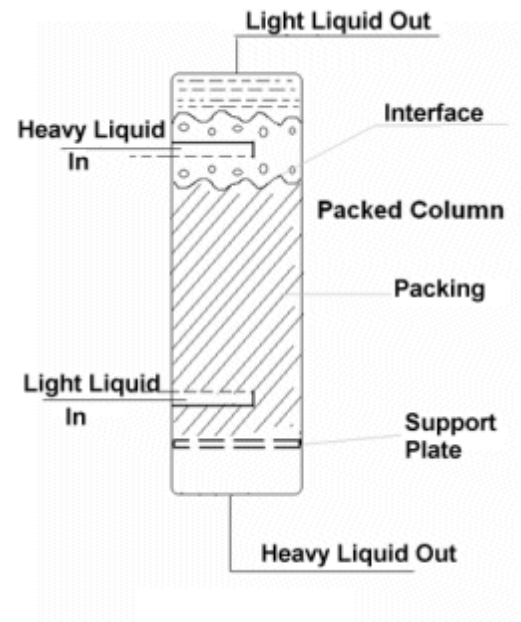
The packed tower is arranged such that the light phase is dispersed. The tower is a cylindrical shell filled with packing which is rested on support plates. The heavy liquid is fed from the top and light liquid is fed from the bottom. A large portion of the void space in the packing is filled with the continuous phase which flows downward. The remainder of the void space is filled with drops of light liquid which rise through the continuous phase and finally coalesce to form a liquid-liquid interface at the top. The packing material should be such that it is wetted by the continuous phase.

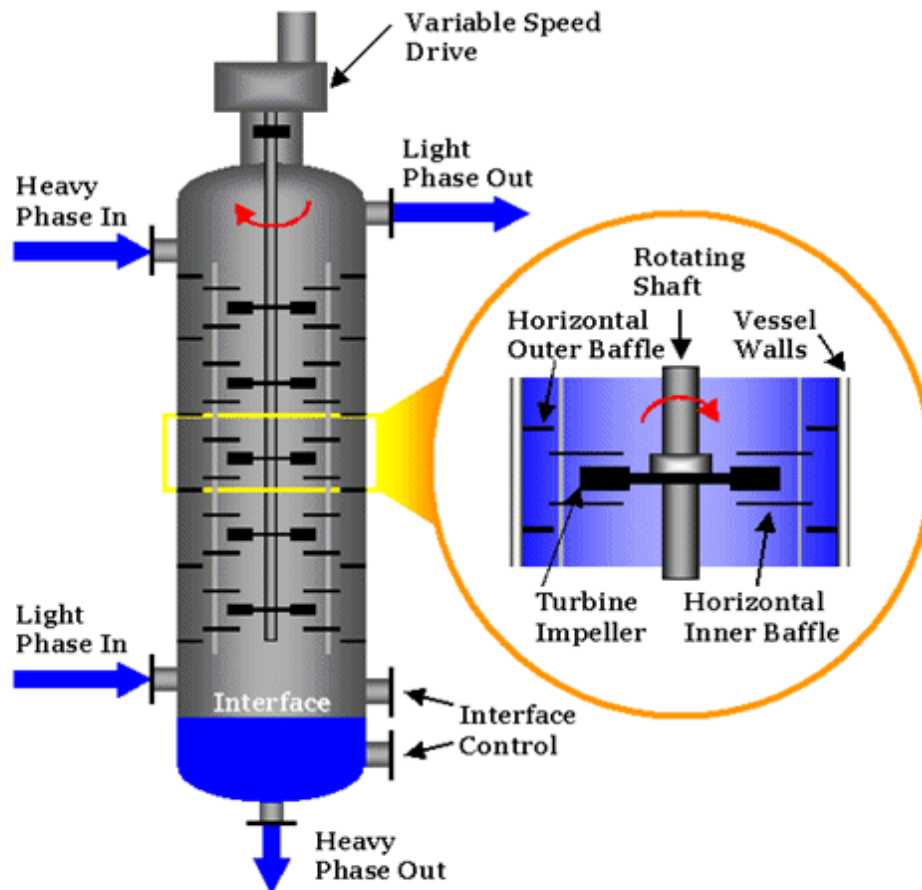
Packing provide large interfacial areas for the phase contacting. This causes the drops to coalesce and reform. The mass transfer rates in packed towers are higher and than those that can be obtained with spray towers because the packing lowers the recirculation of the continuous phase. Packed towers, however, are not appropriate for dirty liquids, suspensions, or highly viscose liquids.

Rotating Disc Contactor

The rotating disc contactor is a mechanically agitated, counter-current extractor. Agitation is supplied by a rotating disc, which usually runs at much higher speeds than a turbine type impeller. The disc contactor consists of a cylindrical column that is divided into number of compartments formed by a series of stator rings. Each compartment contains a centrally located, horizontal rotor disk that creates a high degree of turbulence inside the column. The diameter of the rotor disk is slightly less than the opening in the stationary stator rings. Typically, the disk diameter is 33-66 % of the column diameter. The disk disperses the liquid and forces it outward toward the wall where the stator rings create quite zones where the two phases can separate. Shown below is a Scheibel column which is classified as a type of rotating disc contactor.

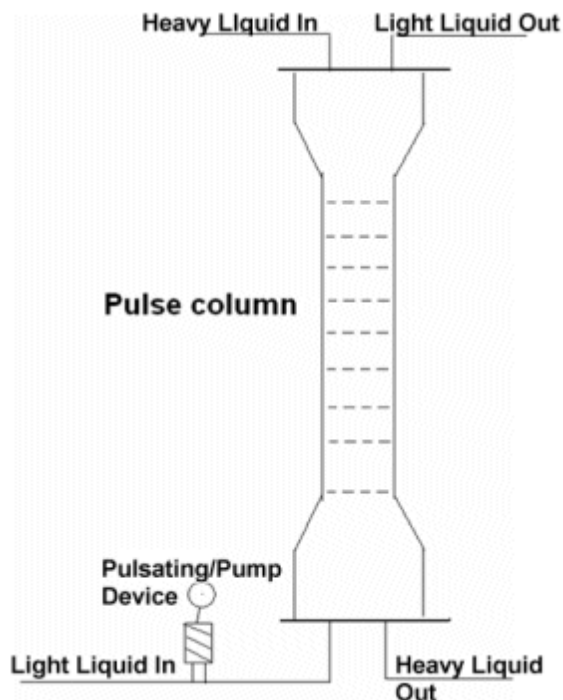
Advantages of the rotating disc contactor include a reasonable capacity, low operating costs, and high efficiency. The main disadvantage is that corrosive liquids can damage the internal moving parts.





Scheibel Column (a type of rotating disk contactor)

Pulse Column

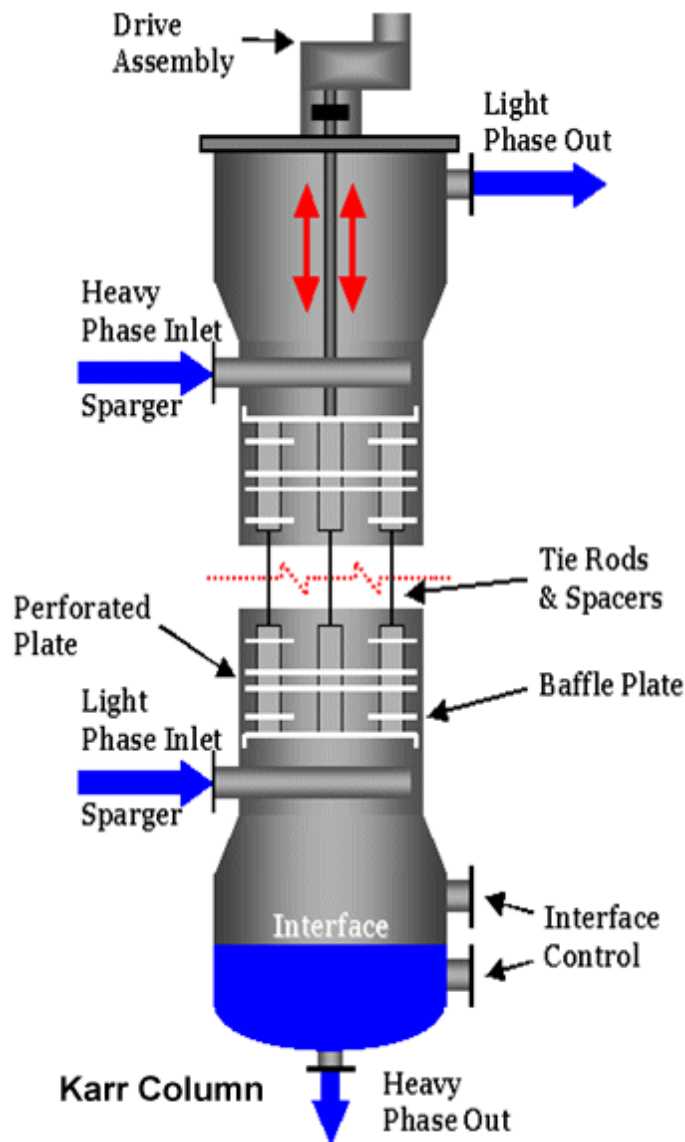


A reciprocating pump is a common mechanical, pulsating device that pulses the entire contents of the column at frequent intervals. The rapid reciprocating motion, of relatively small amplitude, is superimposed on the usual flow of the liquid phases. Bellows, diaphragms made of Teflon-coated steel, or any other reciprocating, pulsating mechanism can be used.

A pulsed, sieve plate column is a vertical column with a large number of sieve plates lacking down comers. The perforations in the plates of such columns are smaller than non-pulsating column (1.5-3.0 mm in diameter). A pulse amplitude of 5-25 mm is generally recommended with a frequency of 100-260 cycles per minute. The pulsation causes the light liquid to be dispersed into the heavy phase on the upward stroke and heavy liquid phase to jet into the light phase on the downward stroke. The column has no moving parts, low axial mixing, and a high extraction efficiency.

A pulse column typically requires less than a third the number of theoretical stages as compared to a non-pulsating column. A pulse column may contain ordinary packing or sieve plates without down comers. A special type of reciprocating mechanism is used in a Karr Column which is shown below.

Pulse columns increase both the turbulence and interfacial areas when compared to other types of columns. This type of column is used extensively for the processing of radioactive solutions.



Centrifugal Contactors

Centrifugal contactors are high-speed, rotary machines with the advantage of a very low residence time. The number of stages in a centrifugal device is usually one, but recently devices with multiple stages are becoming more common. These extractors are mainly used in the pharmaceutical industry. These are ideal for systems in which the density difference is less than 4%. Additionally, this type of system should be utilized if the process requires many equilibrium stages. Centrifugal contactors utilize mechanical devices to agitate the mixture to increase the interfacial area and decrease the mass transfer resistance.

The following table shows the advantages and disadvantages for each type of centrifugal contactor:

Unit of Operation	Advantages	Disadvantages
Mixer-Settler	<ul style="list-style-type: none"> • Efficient • Low head room • Induces good contacting • Can handle any number of stages 	<ul style="list-style-type: none"> • Large floor • High set-up costs • High operation costs
Columns (without agitation)	<ul style="list-style-type: none"> • Small investment costs • Low operating costs 	<ul style="list-style-type: none"> • High head room • Difficult to scale up from lab • Less efficient than mixer-settler
Columns (with agitation)	<ul style="list-style-type: none"> • Good dispersion • Low investment costs • Can handle any number of stages 	<ul style="list-style-type: none"> • Difficult to separate small density differences • Does not tolerate high flow ratios
Centrifugal Extractors	<ul style="list-style-type: none"> • Can separate small density differences • Short holding time • Small liquid inventory 	<ul style="list-style-type: none"> • High set-up cost • High operating and maintenance costs • Cannot handle many stages

Factors Affecting the Selection of Extraction Equipment

When selecting a type of extractor, the following factors should be considered: stage requirements, fluid properties, and operational considerations. The following table outlines the capabilities and characteristics of different types of extractors:

Property	Mixer-Settler	Centrifugal Extractor	Static Columns	Agitated Columns
Number of stages	Low	Low	Moderate	High
Flow rate	High	Low	Moderate	Moderate
Residence time	Very High	Very Low	Moderate	Moderate
Interfacial tension	Moderate to High	Low to Moderate	Low to Moderate	Moderate to High*
Viscosity	Low to High	Low to Moderate	Low to Moderate	Low to High
Density Difference	Low to High	Low to Moderate	Low to Moderate	Low to High
Floor Space	High	Moderate	Low	Low

Reference: Optimize Liquid-Liquid Extraction, R. Madhavan, Cheresources.com

The Karr reciprocating plate extractor can effectively handle low interfacial tension systems. Other factors governing extractor selection include the presence of solids and safety and maintenance requirements.

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Design Criteria

The basic function of extraction equipment is to mix two phases, form and maintain droplets of dispersed phase, and subsequently separate the phases. The following section outlines some of the factors that need to be considered while designing and optimizing extraction equipment.

Mixing

The amount of mixing required is determined by the fluid physical properties such as viscosity, interfacial tension, and the density differences between the two phases. It is important to provide just the right amount of mixing. Less mixing causes the formation of large droplets and decreases interfacial area (interfacial area varies with the square of the droplet diameter). This reduces mass transfer and decreases stage efficiency. Higher agitation (more mixing) minimizes mass transfer resistance during reactions and extraction but contributes to the formation of small droplets or emulsions which are difficult to settle.

In agitated batch extractors, the agitator design is often optimized for a reaction and heat transfer, not extraction, as these are generally multi-purpose vessels.

The agitator imparts maximum energy at the tip where the velocity is highest and minimum energy at the center. This creates non-uniform droplet sizes, with the smallest being formed at the agitator tip. Extraction equilibrium is controlled by the largest droplet size and the smallest droplet controls the settling time. Therefore, excessive agitation sometimes causes difficulties in phase separation. Usually a redesign in terms of configuration or a change in agitation speed helps in optimizing batch time.

Static extraction columns rely completely on the packings or internals and the fluid flow velocities past the internals to create turbulence and droplets. Therefore, these are restricted by minimum flow requirement of at least one of the phases. Agitated columns have more operating flexibility as the specific energy input can be varied in most designs.

Axial mixing (along the column length) in column contactors reduces stage efficiencies. Baffles or similar devices are used



to minimize axial mixing in static as well as agitated columns. It is also important to avoid temperature gradients in columns to prevent thermal currents contributing to axial mixing.

Settling

The settling characteristics depend on the fluid properties (density difference, interfacial tension, and continuous phase viscosity) and the amount of mixing. Stopping the agitator causes settling in agitated batch vessels. In continuous columns, a settling section is provided either as a part of the extractor or as a separate piece of equipment downstream of the extractor.

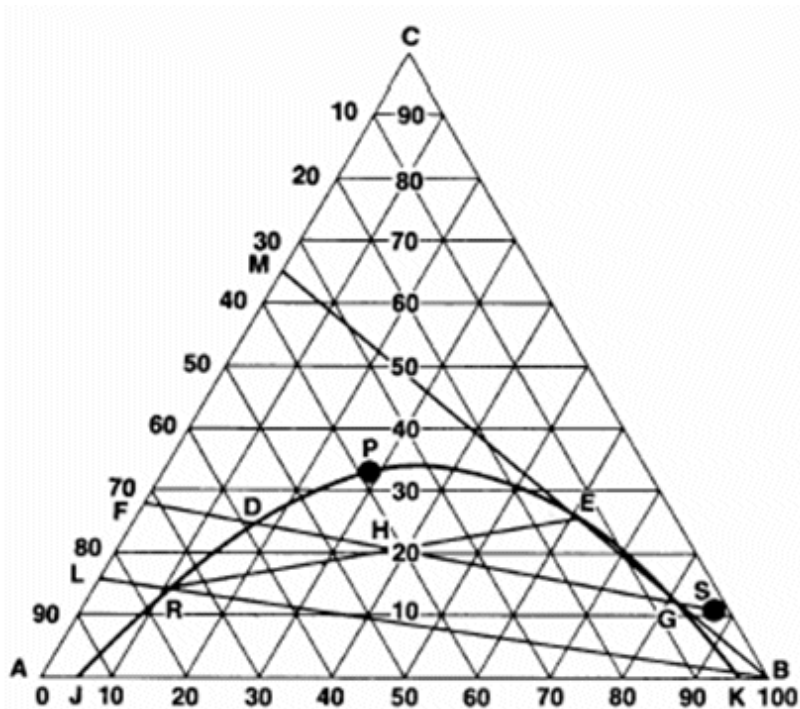
Emulsions are usually formed due to over agitation and in such cases, settling needs to be carried out over an extended period. Emulsions can also form due to the inherent nature of the chemical compounds involved or due to contaminants that substantially lower the interfacial tension. Sometimes coagulants are added to prevent or minimize emulsification. Passing the emulsion layer through a coalescer can break some of these emulsions. In continuous extractors, the creation of emulsions is less severe as good droplet size distribution can be attained at lower agitation speeds. Also, columns such as the Karr reciprocating plate extractor impart uniform energy throughout the radius as a result of the reciprocating motion and this creates a much narrower droplet distribution.

A phenomenon similar to emulsions is the formation of a 'rag layer'. This is a layer containing loose solid substances that float at the interface. These solid substances are generally foreign impurities that exist in the feed streams or those that precipitate from the system during extraction. In continuous extraction, the liquid interface containing the rag layer can be continuously withdrawn, filtered and sent back to the extractor.

Theory - The Ternary Phase Diagram

Ternary phase diagrams act as map for liquid extraction applications. Ternary phase diagrams are unique in that they show all three components of a reaction system on one plot. The following general principles govern ternary phase diagrams:

- Sum of the perpendicular distances from any point within the triangle to the three sides equals the altitude of the triangle.
- Each apex of the triangle represents one of the pure components.
- Any point of a side of the triangle represents a binary mixture.
- Lines may be drawn parallel to the sides of the equilateral triangle for the plotting of the compositions.



Phase Diagram for a Three-Component System

The ternary phase diagram may be constructed directly from experimental data. The saturation curve (miscibility boundary), represented by JDPEK in the figure above, can be obtained experimentally by a cloud point titration. For example, a solution containing components A & C with some composition is made, and then component B is added until the onset of cloudiness due to the formation of a second phase. Then the composition is known for the mixture of the three components and can be plotted onto the ternary phase diagram.

Tie lines are lines that connect points on the miscibility boundary. The tie lines may also be presented on the ternary phase diagram from an experiment. A mixture may be prepared with composition shown by point H (40% A, 40% C, 20% B) from above figure. If allowed to equilibrate, then a chemical analysis of the final extract (E) phase and the raffinate (R) phase can be conducted. Point F is a feed composition into the extractor while point S is the solvent feed to the extractor. Point H represents the composition of the two feeds at equilibrium. This point is determined by summing the feed (F) and solvent (S) compositions for each component. Points R and E are the compositions of the raffinate and extract from the unit, respectively, and the line between them forms the tie line. The tie lines move above and below this line based on the relationship between the raffinate and the extract. Point P represents the plait point. At this point, only one liquid phase exists and the compositions of the two effluents are equal. When C is added to the two phase mixture of A and B, C gets distributed between the phases (layers) and the compositions of the phases follow the raffinate and extract phase solubility curves. Curved line KGEP indicates the composition of the saturated B layer extract phase and the curved line JRDP indicated the composition of the saturated A phase/layer raffinate. The curve represented by JRDPEK is the equilibrium between all three components. The area under the curve is the region where two liquid phases will exist. Above the curve, there will only be one liquid phase. If a line is drawn from F to E or from S to R, this will represent the operating line.

In addition to these considerations, equilibrium constraints must be satisfied.

This implies that:

$$y_{AE} = y_{AR} \quad (1)$$

Where y_{AE} is the activity coefficient for the solute A in the extract and y_{AR} is the activity coefficient of A in the raffinate.

This condition is one of the most important aspects of liquid-liquid extraction since it allows for calculations and assumptions based on equilibrium systems (e.g. the ternary phase diagram).

Another consideration is the separation factor. The goal is to have a separation factor as far from unity as possible. This leads to a better separation in the extraction process. The separation factor is represented as follows:

$$\beta = \frac{y_E^*}{x_R} \quad (2)$$

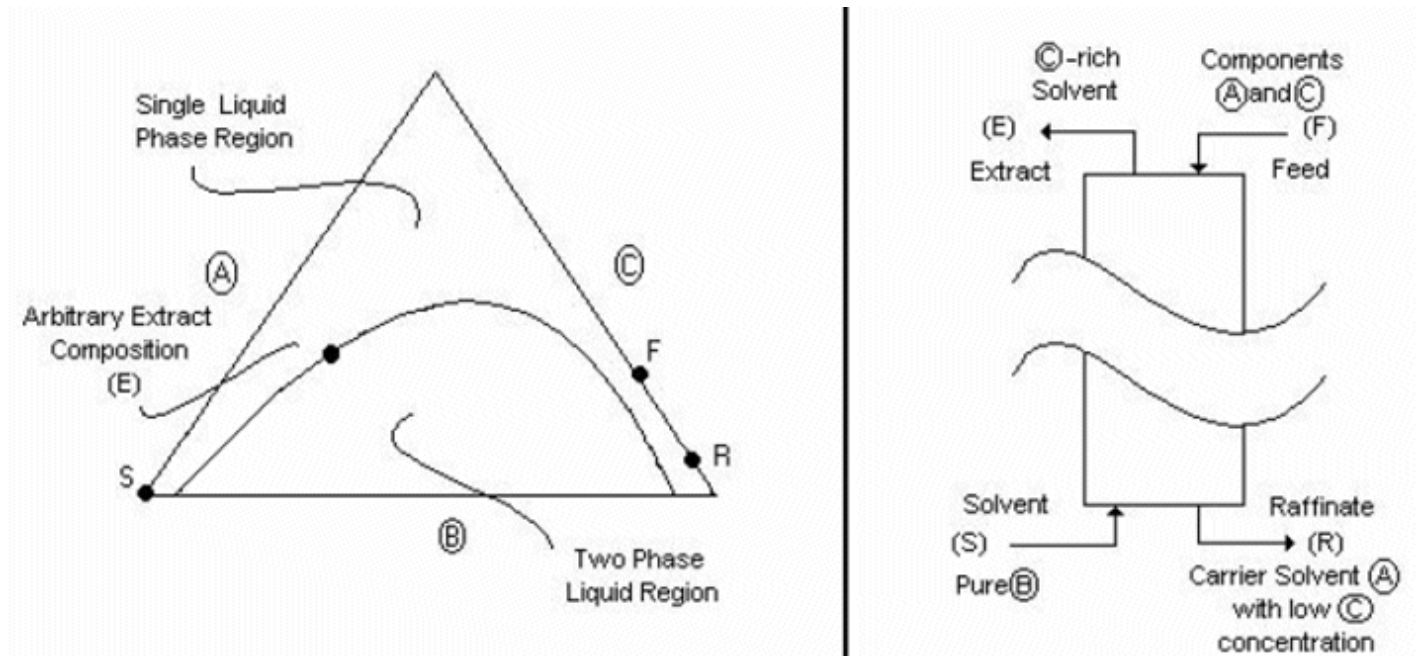
One of the last essential points to the theory behind liquid-liquid extraction is mass transfer. The driving force for this mass transfer arises from the concentration difference between the solute in each of the solvents. In general, it is assumed that the system is at an equilibrium state when mass transfer is occurring. Solute fluxes in the raffinate and extract can be expressed as:

$$N = K_E \cdot A \cdot (x_E^i - x_E) \quad (3)$$

$$N = K_R \cdot A \cdot (x_R^i - x_R) \quad (4)$$

Where K_E and K_R are the overall mass transfer coefficients, A is the cross-sectional area, x_E and x_R are the concentrations of solute in the extract and raffinate respectively, and x_E^i and x_R^i are the concentrations of solute in each phase at the liquid-liquid interface.

The following General Flowchart can be utilized for almost any process. The figure below illustrates a general ternary diagram for a desired solute (C), an extracting solvent (B), and a carrier solvent (A). In this process, it is assumed that the feed (F) contains components A and C. A solvent (S) is introduced in such a way that it will extract C from the feed. The raffinate composition (R) is specified with respect to the recovery of C that is needed.



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