Combine Heat and Mass Transfer Operations

Lec 2: Liquid-Liquid Extraction-part 2

Content

Extraction Equipments

Prof. Zayed Al-Hamamre

Content

- Extraction Equipment

Principal references: Chapter 12 in C.J. Geankoplis book and Chapter 8 in Henley, Seader & Roper book

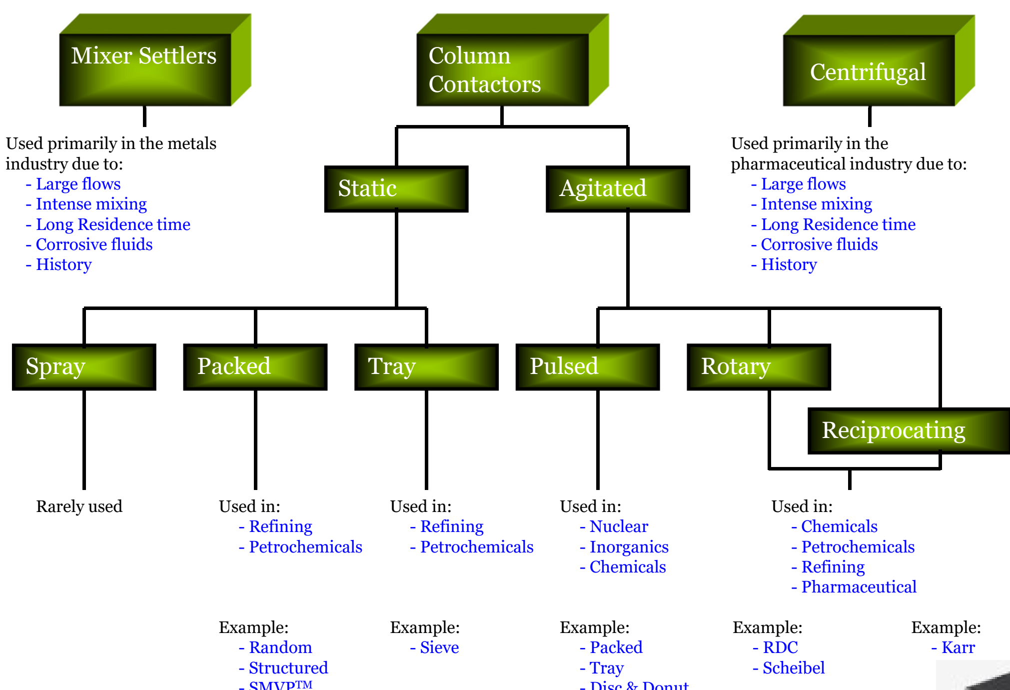
Extraction Equipment

- Different mechanical devices are used in liquid-liquid extraction such as:

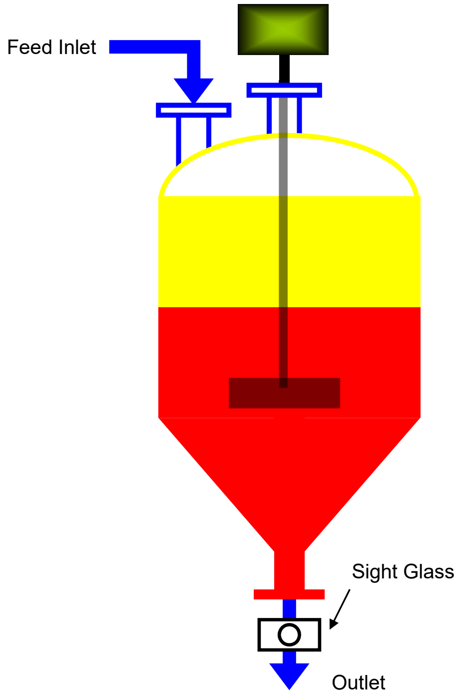
  1. The simplest is a mixer/settler, or decanter, in which the two liquid phases are separated.
  2. Plate towers, packed towers, and mechanically agitated mixers (rotating disk contactors)

- Concept of operation: Batchwise or continuous operation
  - Feed liquid + solvent (put in agitated vessel) = layers (to be settled and separated)
  - Extract – the layer of solvent + extracted solute
  - Raffinate – the layer from which solute has been removed
  - Extract may be lighter or heavier than raffinate.
- Continuous flow – more economical for more than one contact process

Major Types of Extraction Equipment



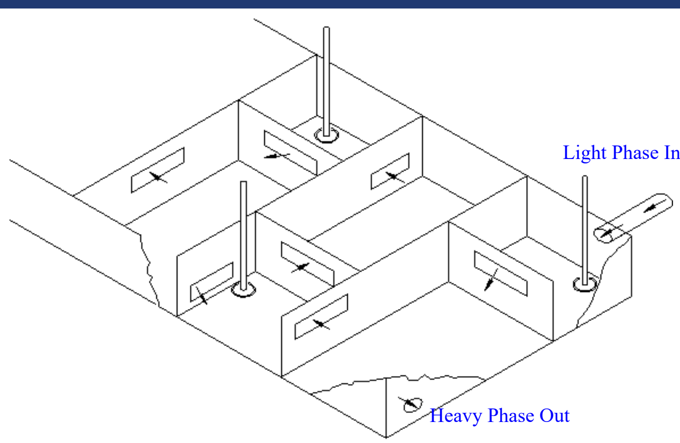
Mix / Decant Tank

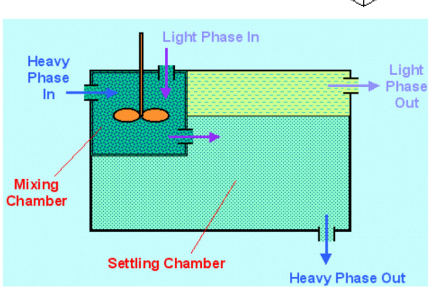


Characteristics

- Mix – Settle – Phase separate in a single tank
- Batch Processing only
- Requires multiple solvent additions for more than one stage (crossflow operation)
- Typically used for small capacity operations or intermittent processing

Mixer / Settlers





Characteristics

- Handle very high flowrates
- Good for processes with relatively slow reactions (residence time required)
- Provide intense mixing to promote mass transfer
- Require large amount of floor space
- Suitable when few theoretical stages required
- Large solvent inventory (and losses)

Mixer / Settlers

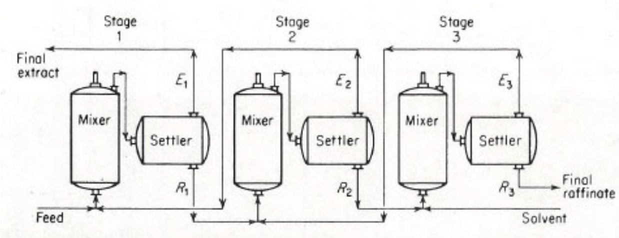


Figure 10.41 Flowsheet of three-stage countercurrent mixer-settler extraction cascade.

Treybal (1980)

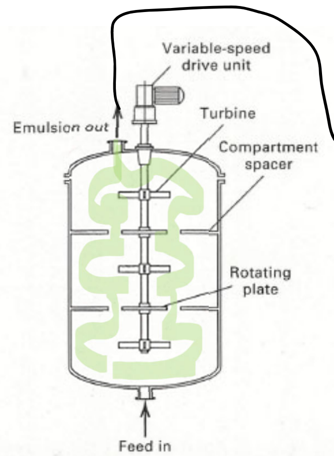


Figure 8.2 Compartmented mixing vessel with variable-speed turbine agitators.

[Adapted from R.E. Treybal, *Mass Transfer*, 3rd ed., McGraw-Hill, New York (1980).]

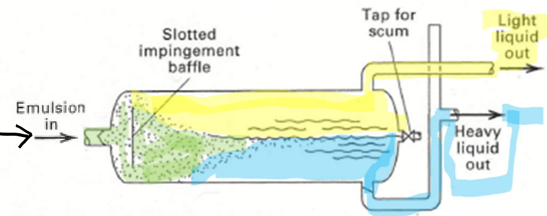


Figure 8.4 Horizontal gravity-settling vessel.

[Adapted from R.E. Treybal, *Liquid Extraction*, 2nd ed., McGraw-Hill, New York (1963) with permission.]

Seader & Henley (2006)

Centrifugal Extractor

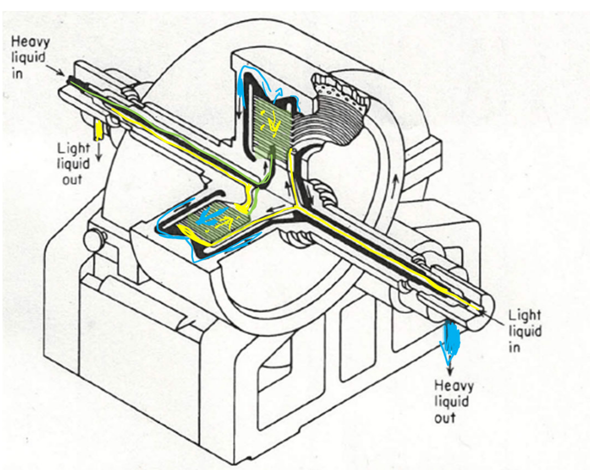


Figure 10.56 Podbielniak centrifugal extractor (schematic). *(Podbielniak, Inc.)*

Characteristics

- Countercurrent flow via centrifugal force
- Low residence time ideally suited for some pharmaceutical applications
- Handles low density difference between phases
- Provide up to several theoretical stages per unit
- High speed device requires maintenance
- Susceptible to fouling and plugging due to small clearances

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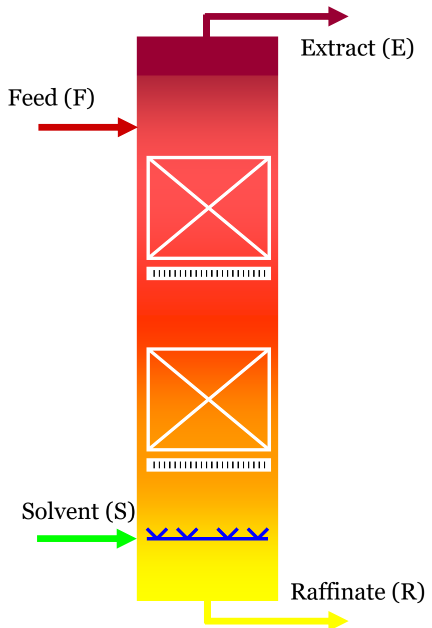
Packed Column



Characteristics

- High capacity:
  20-30 M^3/M^2-hr (Random)
  500-750 gal/ft^2-hr (Random)
  40-80 M^3/M^2-hr (Structured)
  1,000-2,000 gal/ft^2-hr (Structured)
- Poor efficiency due to backmixing and wetting
- Limited turndown flexibility
- Affected by changes in wetting characteristics
- Limited as to which phase can be dispersed
- Requires low interfacial tension for economic usefulness
- Not good for fouling service

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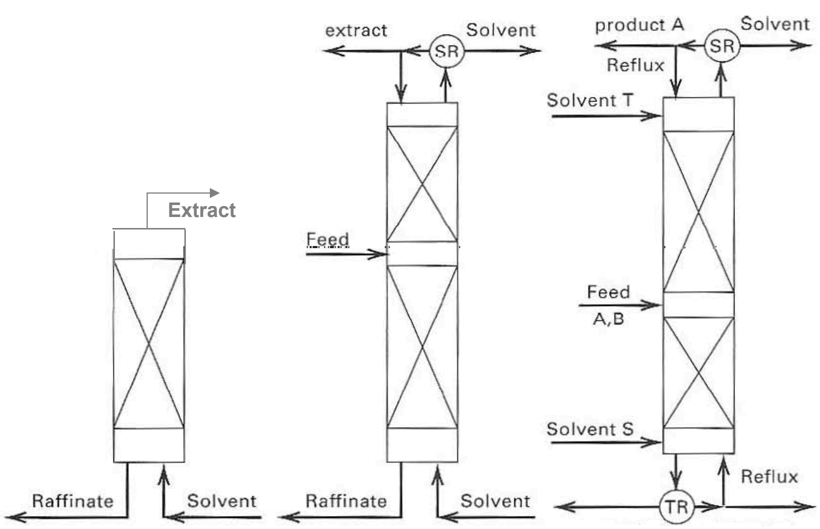
Packed Column



Single-section cascade

Two-section cascade

Dual solvent with two-section cascade

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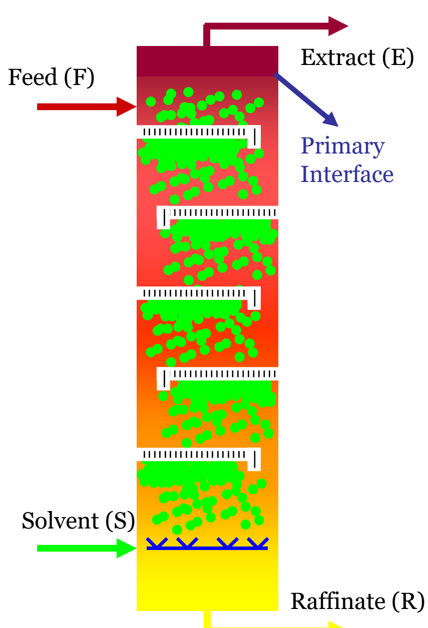
Packed Column

Non-Structured

Structured

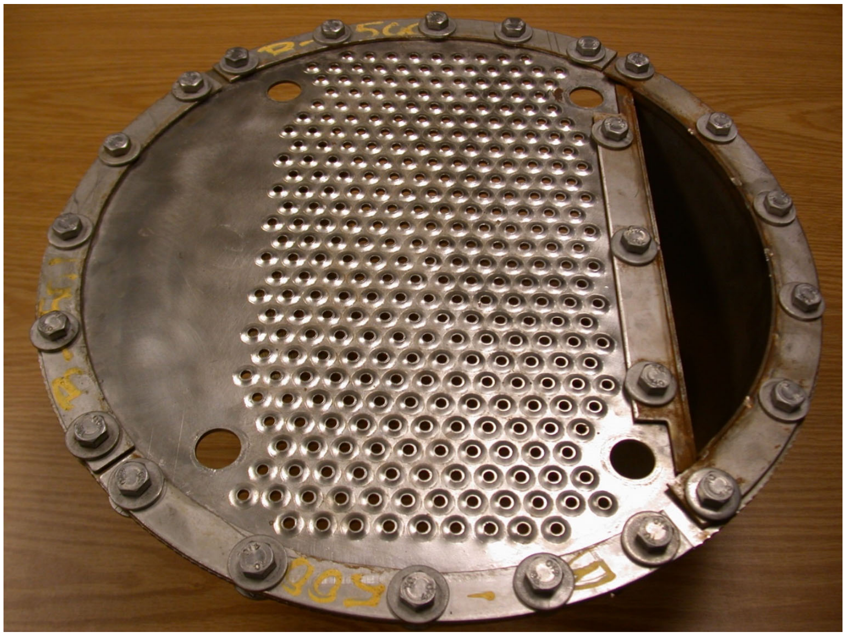
Sieve Tray Column



Characteristics

- High capacity: 30-50 M^3/M^2-hr
  750-1,250 gal/ft^2-hr
- Good efficiency due to minimum backmixing
- Multiple interfaces can be a problem
- Limited turndown flexibility
- Affected by changes in wetting characteristics
- Limited as to which phase can be dispersed

Sieve Tray Column



Extractor Sieve Tray

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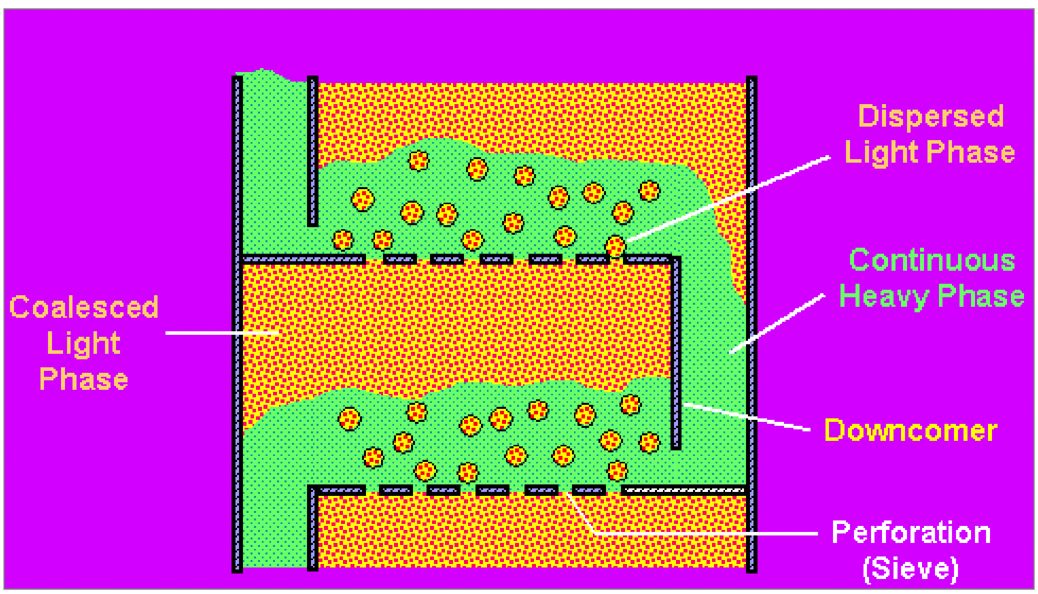
Sieve Tray Column



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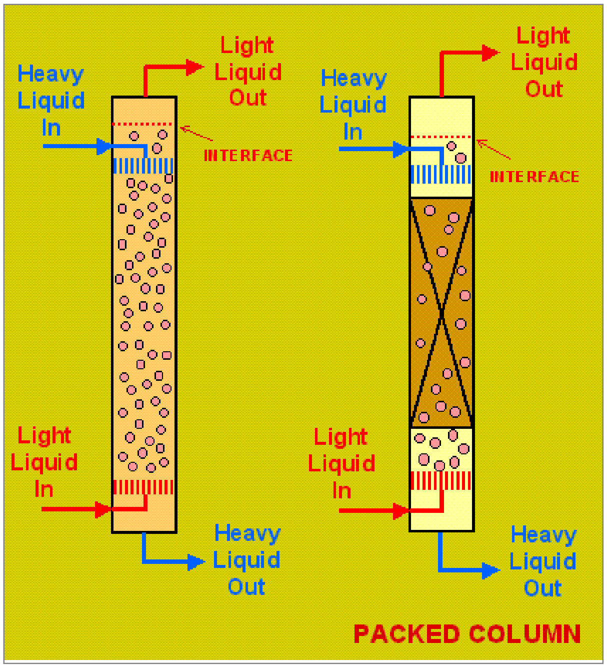
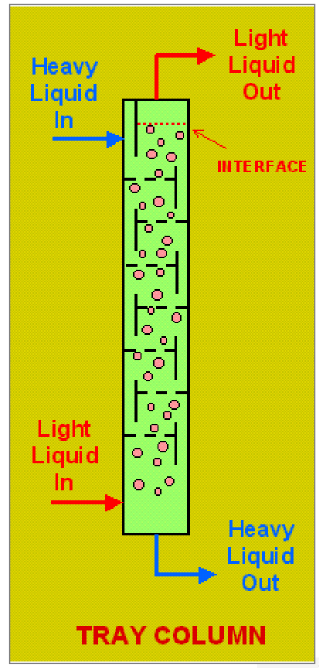
Spray column





Spray column

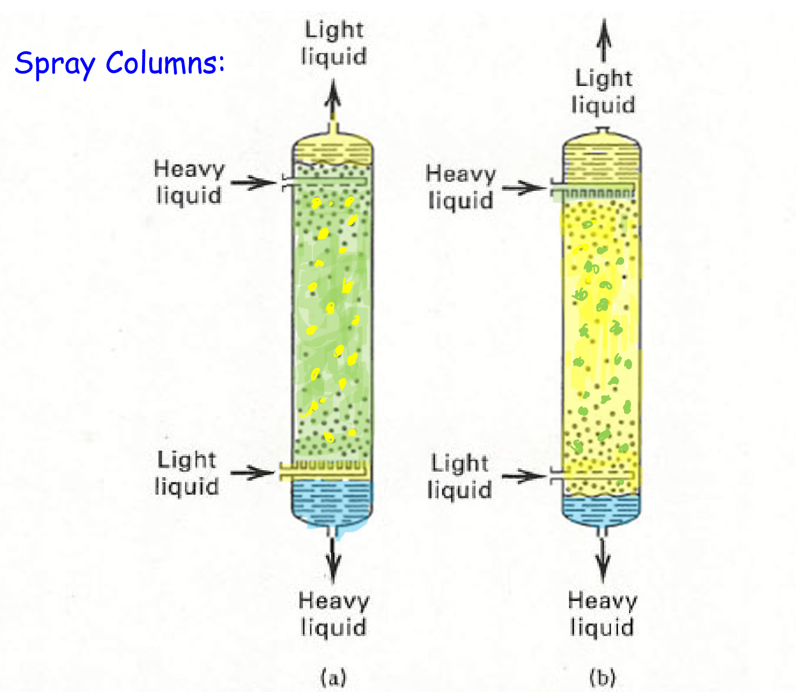
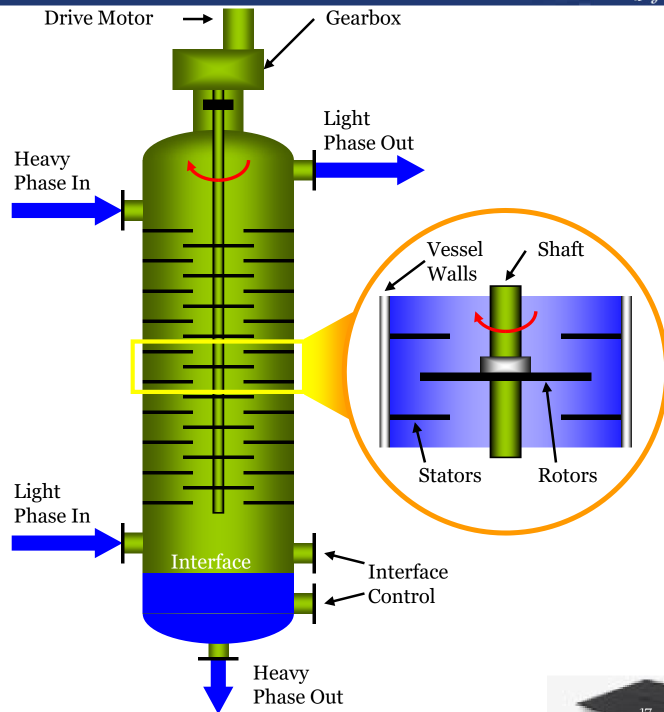


Figure 8.5 Spray columns: (a) light liquid dispersed, heavy liquid continuous; (b) heavy liquid dispersed, light liquid continuous.

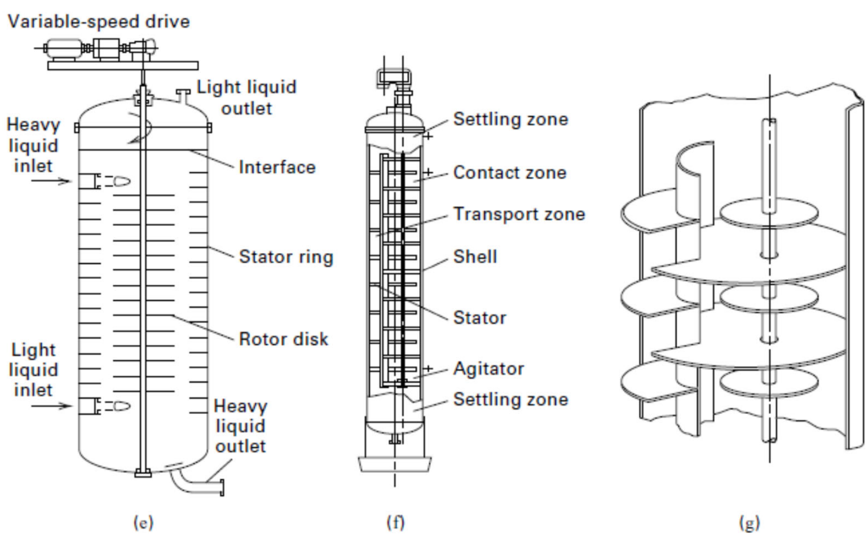
Rotating Disk Contractor (RDC) Extractor

Characteristics

- Reasonable capacity: 20-30 M^3/M^2-hr
- Limited efficiency due to axial backmixing
- Suitable for viscous materials
- Suitable for fouling materials
- Sensitive to emulsions due to high shear mixing
- Reasonable turndown (40%)



Rotating Disk Contractor (RDC) Extractor

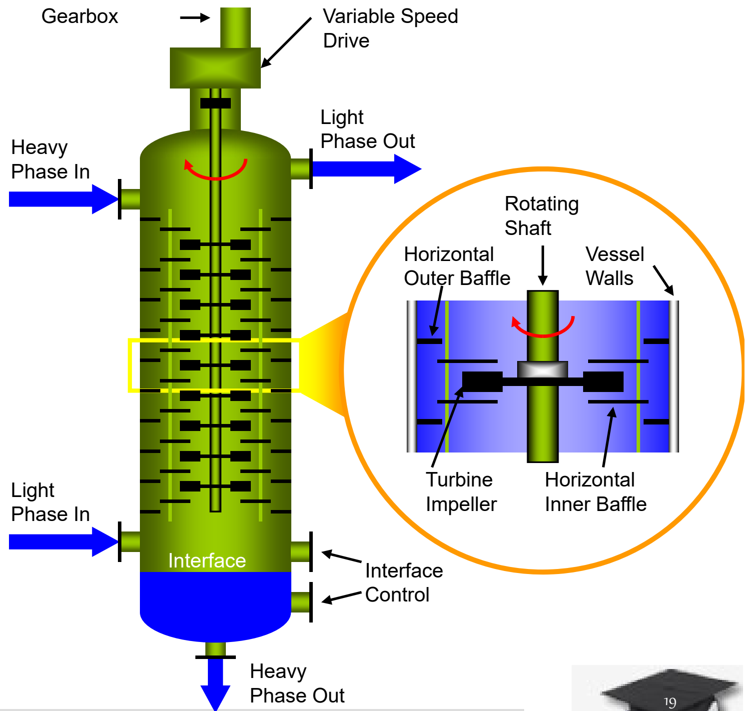


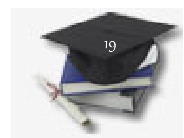
(e) rotating-disk-contactor (RDC);
(f) asymmetric rotating-disk contactor
(ARD); (g) section of ARD contactor;

Scheibel Column

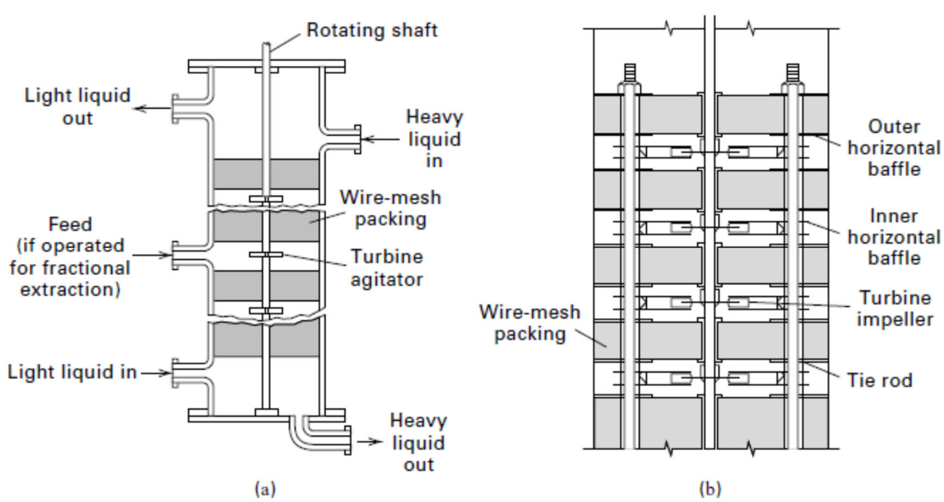
Characteristics

- Reasonable capacity:
  15-25 M^3/M^2-hr
  350-600 gal/ft^2-hr
- High efficiency due to internal baffling
- Good turndown capability (4:1) and high flexibility
- Best suited when many stages are required
- Not recommended for highly fouling systems or systems that tend to emulsify





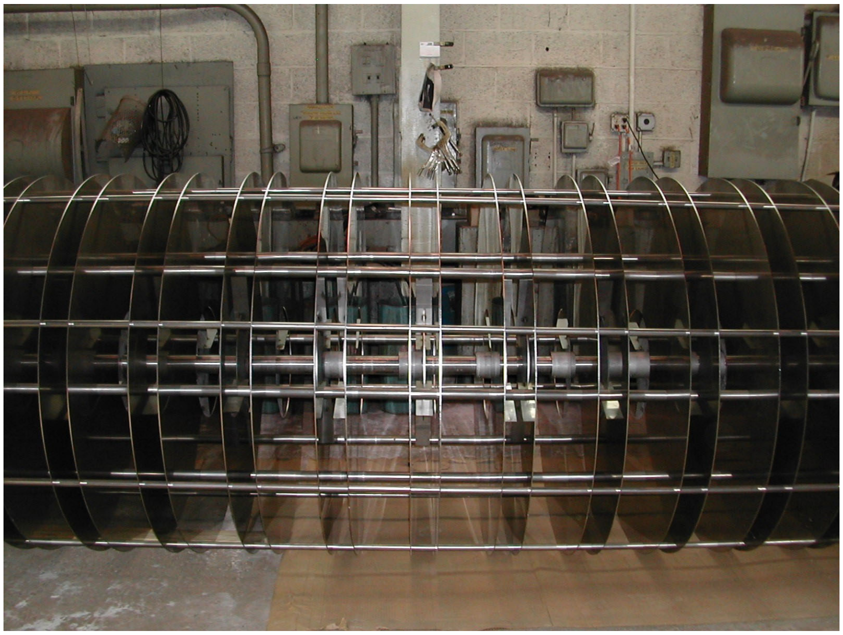
Scheibel Column



Columns with Mechanically Assisted Agitation

Figure 8.7 Commercial extractors with mechanically assisted agitation:
(a) Scheibel column—first design;
(b) Scheibel column—second design;

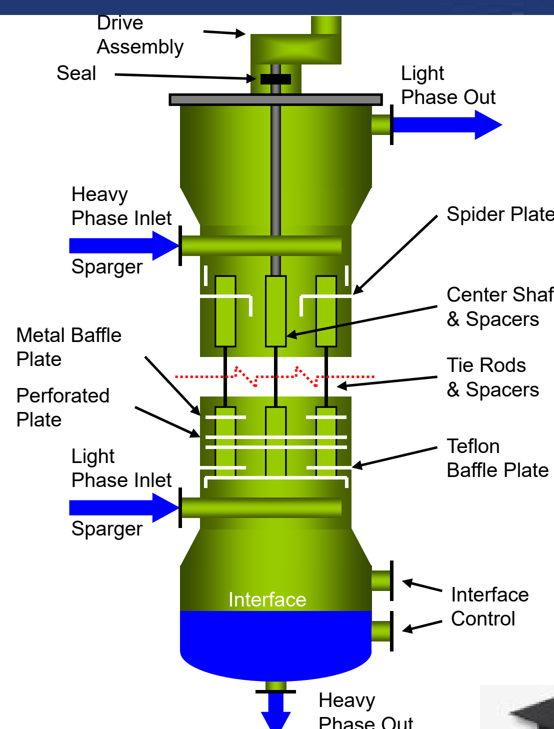
Scheibel Column Internal Assembly



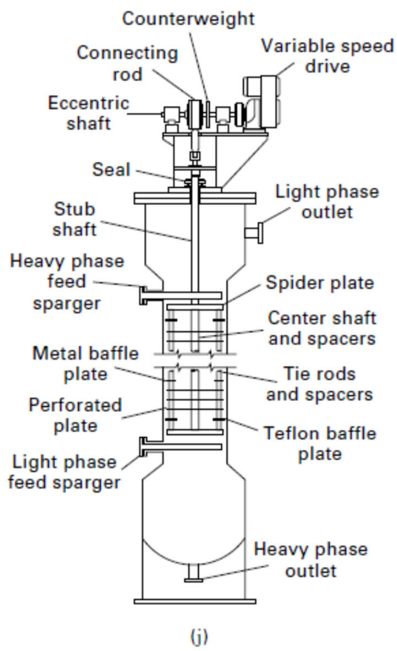
Karr Reciprocating Column

Characteristics

- Highest capacity:
  30-60 M^3/M^2-hr
  750-1,500 gal/ft^2-hr
- Good efficiency
- Good turndown capability (4:1)
- Uniform shear mixing
- Best suited for systems that emulsify



Karr Reciprocating Column



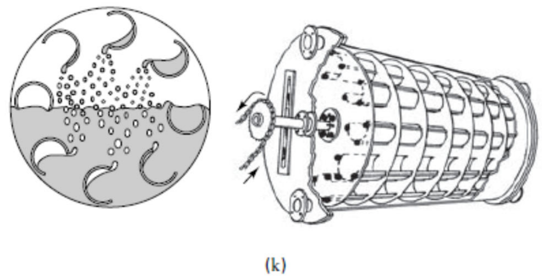


Figure 8.7 (*Continued*) (j) Karr reciprocating-plate column (RPC); (k) Graesser raining-bucket (RTL) extractor.

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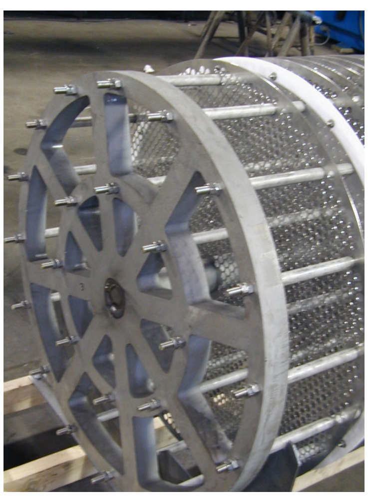
Karr Column Plate Stack Assembly



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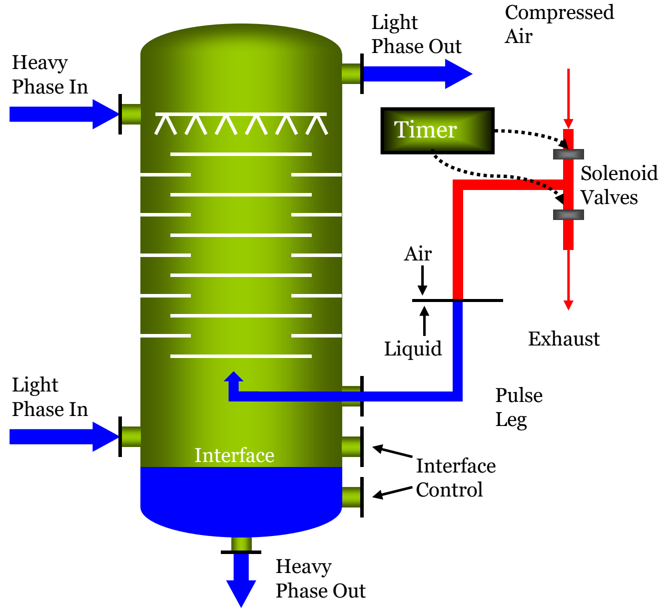
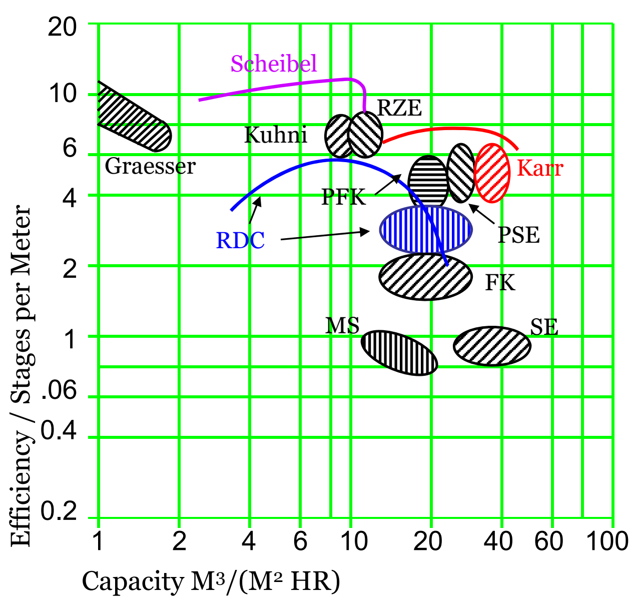
Pulsed Extractor

Characteristics

- Reasonable capacity:
  20-30 M^3/M^2-hr
- Best suited for nuclear applications due to lack of seal
- Also suited for corrosive applications since can be constructed out or non-metals
- Limited stages due to backmixing
- Limited diameter/height due to pulse energy required



Comparison Plot of Various Commercial Extractors



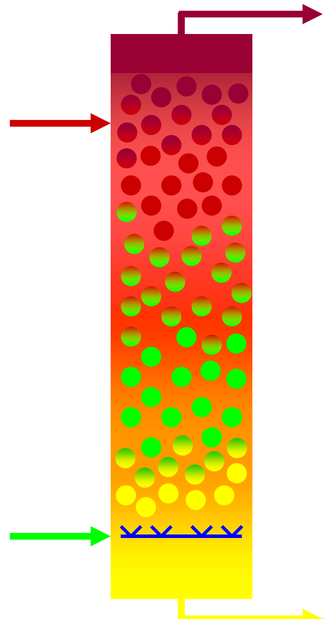
Key

- Graesser = Raining Bucket
- MS = Mixer Settler
- SE = Sieve Plate
- FK = Random Packed
- PFK = Pulsed Packed
- PSE = Pulsed Sieve Plate
- RDC = Rotating Disc Contactor
- RZE = Agitated Cell
- Karr = Karr Recipr. Plate
- Kuhni = Kuhni Column
- Scheibel = Scheibel Column

Column Selection Criteria
Static Column

A static column design may be appropriate when:

- Interfacial tension is low to medium: up to 10-15 dynes/cm
- Only a few theoretical stages are required, and reduction in S/F is not an economic benefit
- No operational flexibility required
- There is a large difference in solvent to feed rates



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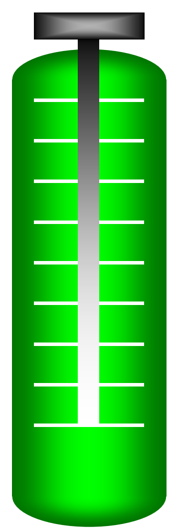
Column Selection Criteria
Agitated Column

Agitated columns are generally more economical when:

- More than 2-3 theoretical stages are required
- Interfacial tension is moderate to high, although low interfacial tensions may also be economical
- A reduction in solvent usage is beneficial to the process economics
- The process requires a wide turndown as well as the ability to handle a range of S/F ratios



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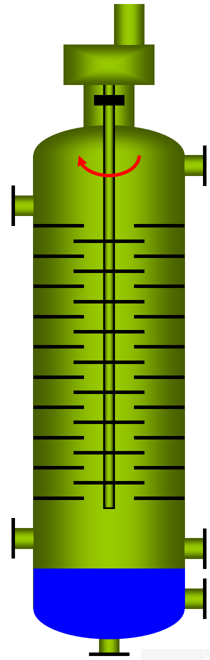
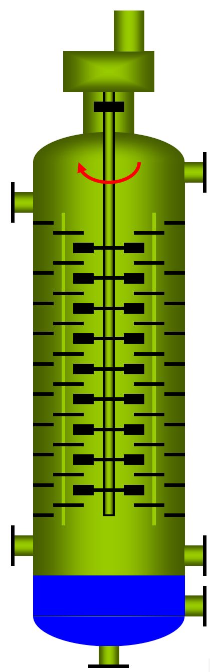
Column Selection Criteria
Rotating Disc Contactor (RDC)

- Systems with moderate to high viscosity, i.e. > 100 cps
- Systems that are residence time controlled, for example, slow mass transfer rate with few theoretical stages required
- Systems with a high tendency towards fouling



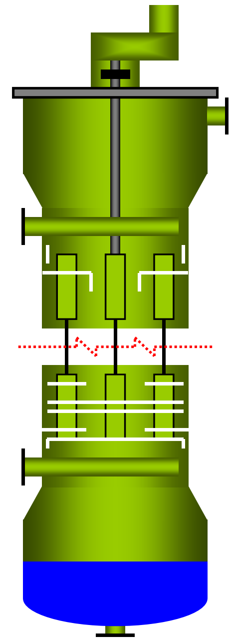
Column Selection Criteria
Scheibel Column

- Systems that require a large number of stages due to either theoretical stage requirements or low mass transfer rates
- Low volume applications in which a relatively small column is required
- Systems that process relatively easily, without a tendency to emulsify and/or flood



Column Selection Criteria
Karr Reciprocation Plate Column

- Difficult systems that tend to emulsify and/or flood easily
- Systems in which the hydraulic behavior varies significantly through length of the column
- Sometimes requiring non-metallic internals, such as Teflon due to wetting characteristics or corrosive materials
- Fouling applications that may have tars formations and/or solids precipitation



The Three Cornerstones of Successful Extraction Applications

Successful Application

Proper Solvent Selection

Selection Based on:

- Sound thermodynamic principles
- Sound economic principles
  - Availability
  - Recoverability
- Sound environmental principles
  - Toxicity
  - Safety

Meaningful Pilot Tests

Testing Based on:

- Actual feed stocks
- Full process including solvent recovery
- Wide range of operating conditions

Accurate Scale-Up

Scale-Up Based on:

- Proven techniques
- Proper safety factors

Table 8.3 Advantages and Disadvantages of Different Extraction Equipment

Class of Equipment	Advantages	Disadvantages
Mixer-settlers	Good contacting Handles wide flow ratio Low headroom High efficiency Many stages available Reliable scale-up	Large holdup High power costs High investment Large floor space Interstage pumping may be required
Continuous, counterflow contactors (no mechanical drive)	Low initial cost Low operating cost Simplest construction	Limited throughput with small density difference Cannot handle high flow ratio High headroom Sometimes low efficiency Difficult scale-up
Continuous, counterflow contactors (mechanical agitation)	Good dispersion Reasonable cost Many stages possible Relatively easy scale-up	Limited throughput with small density difference Cannot handle emulsifying systems Cannot handle high flow ratio
Centrifugal extractors	Handles low-density difference between phases Low holdup volume Short holdup time Low space requirements Small inventory of solvent	High initial costs High operating cost High maintenance cost Limited number of stages in single unit

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Table 8.2 Maximum Size and Loading for Commercial Liquid–Liquid Extraction Columns

Column Type	Approximate Maximum Liquid Throughout, m^3/m^2-h	Maximum Column Diameter, m
Lurgi tower	30	8.0
Pulsed packed	40	3.0
Pulsed sieve tray	60	3.0
Scheibel	40	3.0
RDC	40	8.0
ARD	25	5.0
Kuhni	50	3.0
Karr	100	1.5
Graesser	<10	7.0

Above data apply to systems of:

1. High interfacial surface tension (30 to 40 dyne/cm).
2. Viscosity of approximately 1 cP.
3. Volumetric phase ratio of 1:1.
4. Phase-density difference of approximately 0.6 g/cm^3.

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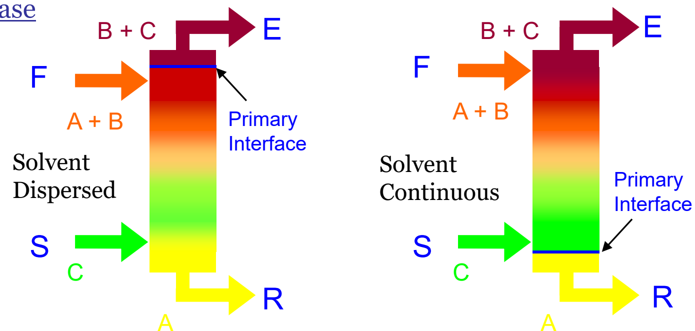
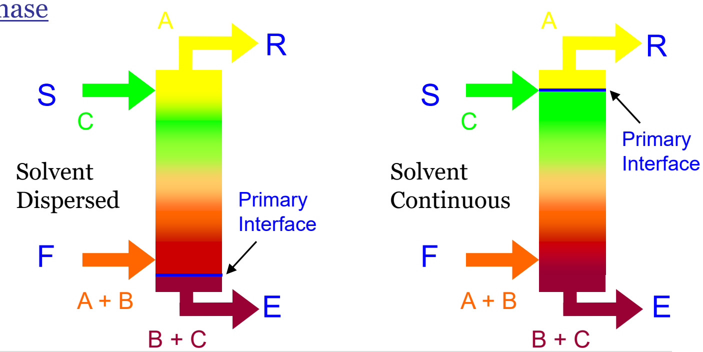
Possible Extraction Column Configurations

Solvent is Light Phase



Solvent is Heavy Phase



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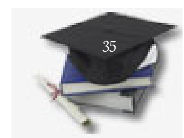


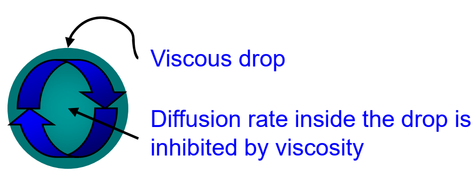
Factors Effecting which Phase is Dispersed

Flow Rate

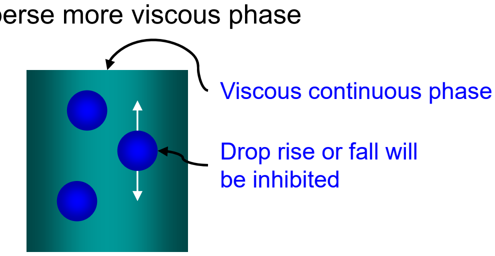
- For Sieve Tray and Packed Columns – disperse the higher flowing phase
- For all other columns – disperse lower flowing phase

Viscosity

- For efficiency – disperse less viscous phase



- For capacity – disperse more viscous phase



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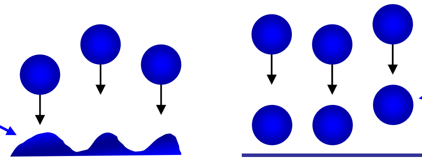
Factors Effecting which Phase is Dispersed

Surface Wetting

- Want the continuous phase to preferentially set the internals – this minimizes coalescence and therefore maximizes interfacial area.

Droplets coalesce.
Interfacial area lost.



Droplets retain shape.
Maximizes interfacial area.

Importance of maintaining droplets

Assume – 30% holdup of dispersed phase in 1 M^3 of solution

Droplet Diameter [μ]	Droplet Volume [M^3]	Number Droplets	Droplet SA [M^2]	Interfacial Area [M^2/M^3]
100	0.3	7.16×10^{10}	1.26×10^{-7}	9022
300	0.3	2.65×10^{9}	1.13×10^{-6}	2995
500	0.3	5.73×10^{8}	3.14×10^{-6}	1796

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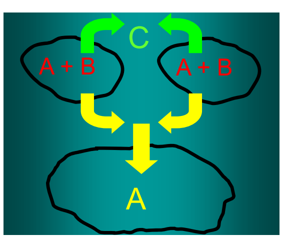
Factors Effecting which Phase is Dispersed

Marangoni Effect

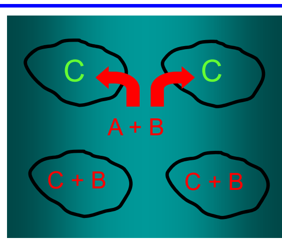
- Coalescence is enhanced by mass transfer from droplets → continuous phase



Mass Transfer Direction

Dispersed → Continuous
(d → c)

- Droplets tend to coalesce
- Must be counteracted by additional energy



Continuous → Dispersed
(c → d)

- Droplets tend to repel each other
- Less energy required to maintain dispersion

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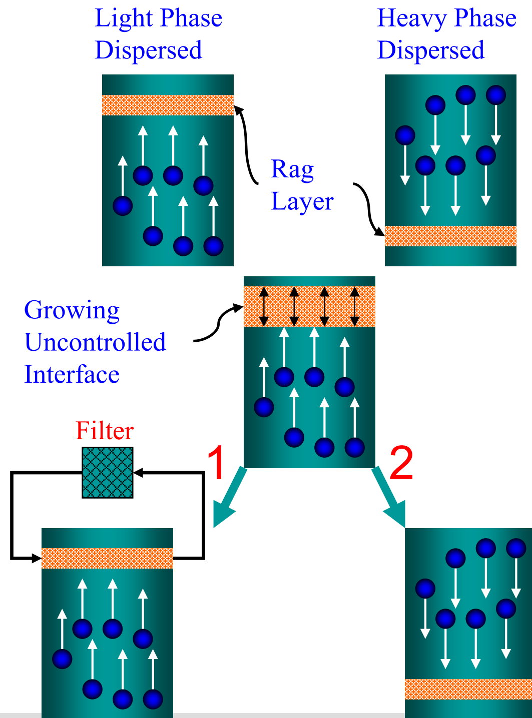
Interface Behavior

Actions to control unstable interface

As extraction proceeds, interface normally grows in thickness and forms a "rag" layer that stabilizes at some thickness

If rag layer continues to grow, some action must be taken

1. Rag Draw
   Continuously withdraw a portion of the interface and pass through a filter to remove interfacial contamination
2. Reverse Phases
   Often a stable interface can be controlled by reversing which phase is dispersed

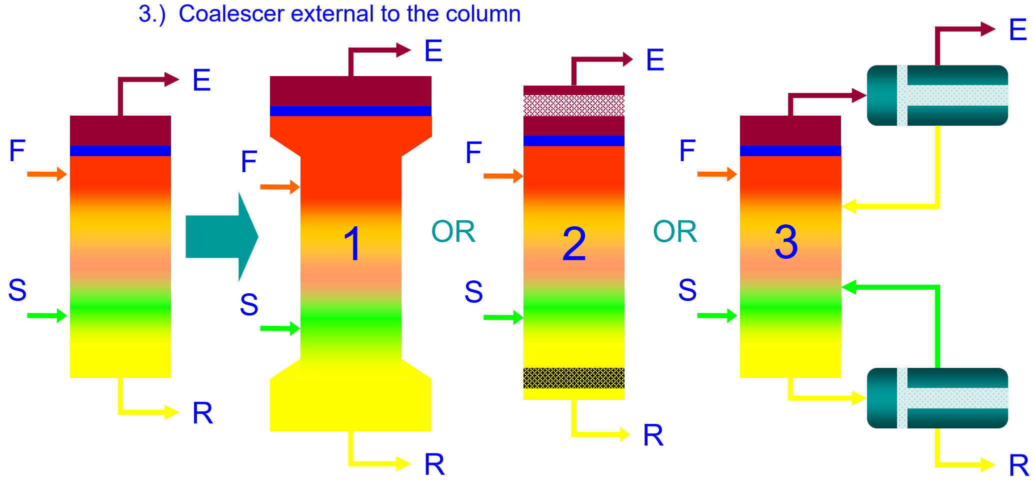


Entrainment

Entrainment involves carrying over a small portion of one phase out the wrong end of the column.

Entrainment is controlled by:

1.) Increased settling time inside the column
2.) Coalescer inside the column
3.) Coalescer external to the column

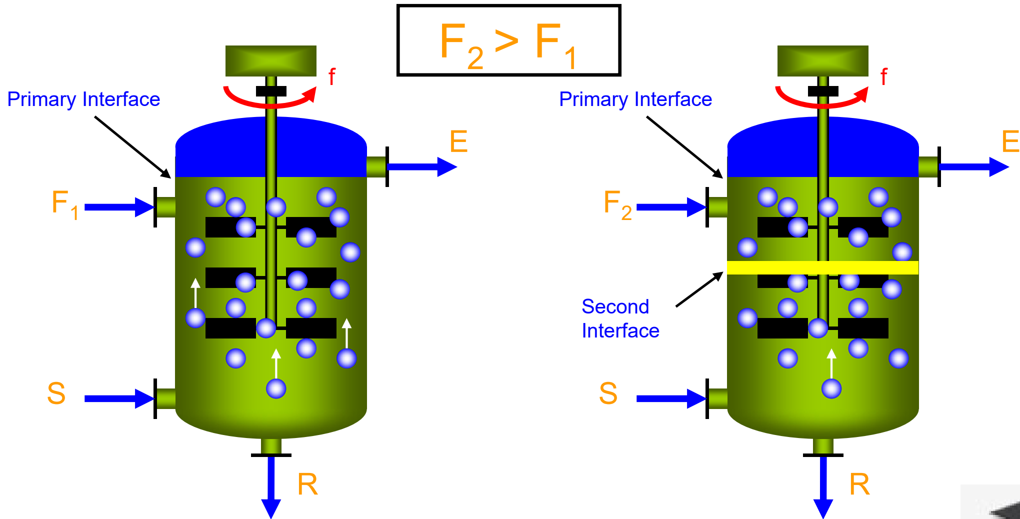


Flooding

Flooding – the point where the upward or downward flow of the dispersed phase ceases and a second interface is formed in the column.

Flooding can be caused by:

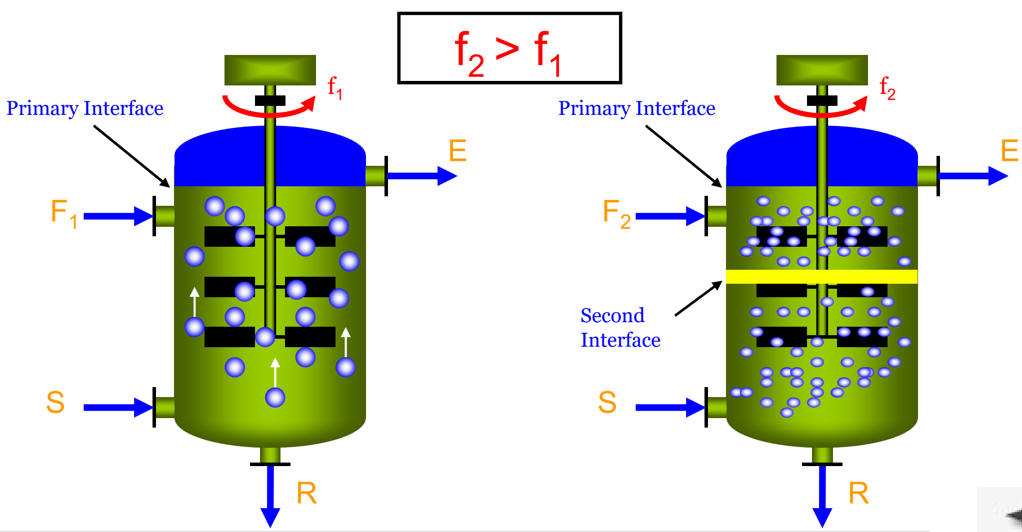
- Increased continuous phase flow rate which increases drag on droplets



Flooding

Flooding can be caused by:

- Increased agitation speed which forms smaller droplets which cannot overcome flow of the continuous phase
- Decreased interfacial tension – forms smaller drops – same effect as increased agitation



Safety concerns

Liquid-liquid (solvent) extraction units safety concerns can be reduced by:

- Using a high-ash point solvent
- Avoid static electricity generation (sparking)
- Slow liquid ow rates in pipes < 1 m/s to avoid static build-up
- Use conductive piping, not plastics or rubbers
- Feed organic phase from the bottom of tank, not the side, to avoid splashing
- Avoid any areas for air pockets to form
- Avoid generating mists of solvent
- Electrical circuits must be rated explosion proof
- Strict rules related to mobile electronics, welding, etc in the neighbourhood
- Conduct full HAZOP and re assessment
- Do not allow trenches/dips for the solvent phase to accumulate, when spills occur