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Process Chem and Pharmaceutical Engineering 1

Liquid-liquid Separation Part 1

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Liquid-liquid Separation Part 1

By Wan Nurul Huda

<http://ocw.ump.edu.my/course/view.php?id=350#section-5>

Chapter Description

- Aims
 - Solve problems related to extraction process by applying the formula relevant to specific unit operations
- Expected Outcomes
 - Describe the concept of equilibrium between two liquids phases
- References
 - Unit Operations of Chemical Engineering, Warren L. McCabe, Julian C. Smith, Peter Harriott



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Introduction to Extraction

- Liquid-liquid extraction (also known as solvent extraction) involves the separation of the constituents (solutes) of a liquid solution by contact with another insoluble liquid.
- Solutes are separated based on their different liquids.
- Separation is achieved when the substances constituting the original solution is transferred from the original solution to the other liquid solution.

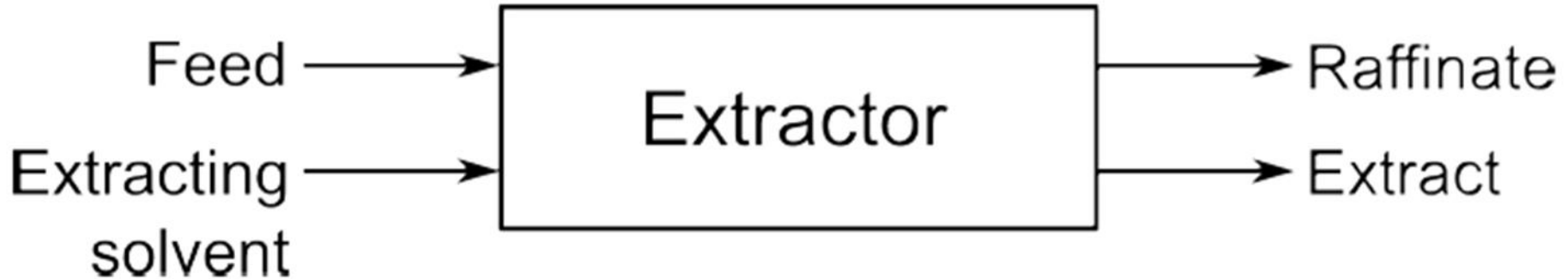


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Definition



- Solute: species we aim to recover (A) from the feed
- Feed or feed solvent: one of the liquids in the system (carrier)
- Solvent: MSA (by convection: the added liquid)
- Extract: solvent (not solute) mostly present in this layer
- $y_{E,A}$ = concentration of A, the solute in extract
- Raffinate: residual solute in this layer = $x_{R,A}$



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Solvent Extraction (Partition Coefficient)

- The distribution of solute between two phases is expressed quantitatively by distribution or partition coefficient, K_D .

$$K_D = \frac{\textit{solute concentration in extract phase}}{\textit{solute concentration in raffinate phase}}$$

- Higher value of K_D indicates higher extraction efficiency



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- The simplest liquid-liquid extraction involves only a ternary (i.e. 3 components) system.
- The solution which is to be extracted is called the feed, and the liquid with which the feed is contacted is the solvent.
- The feed can be considered as comprising the solute A and the carrier liquid C. Solvent S is a pure liquid.
- During contact, mass transfer of A from the feed to the solvent S occurs, with little transfer of C to S.
- The solvent (with the solute) is then permitted to separate from the carrier liquid.



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- The solvent-rich product of the operation is called the extract, and the residual liquid from which solutes has been removed is the raffinate
- In some operations, the solutes are the desired product, hence the extract stream is the desirable stream.
- In other applications, the solutes may be the contaminants that need to be removed, and in this instance the raffinate is the desirable product stream.

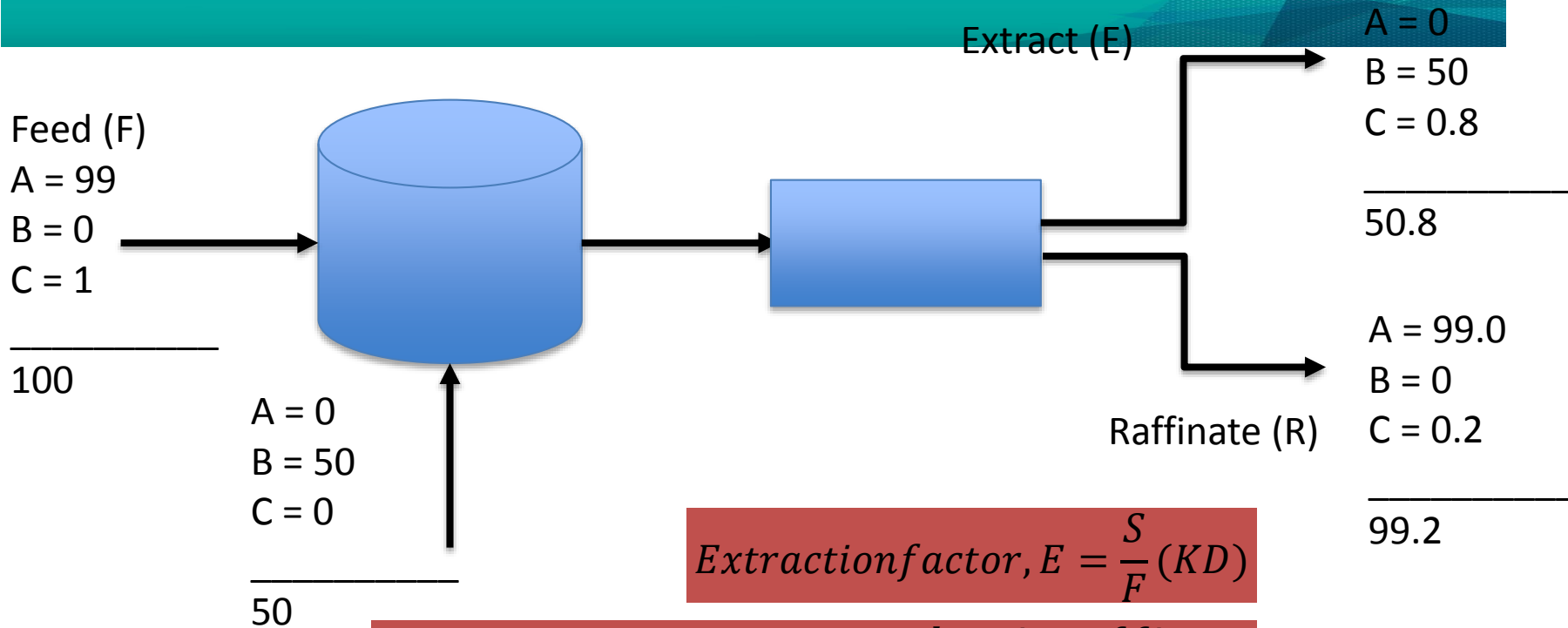


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Simple Extraction Single Stage



$$\text{Extraction factor, } E = \frac{S}{F} (KD)$$

$$\text{Fraction unextracted, } U = \frac{\text{solute in raffinate}}{\text{solute in feed}}$$

$$\text{Distribution coefficient, } K_D = \frac{\text{conc. solute in extract}}{\text{conc. solute in raffinate}}$$



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Where LLE is normally used?

- Bioseparations
- Nuclear (uranium recovery)
- Mining: nickel/cobalt
- Perfumes, fragrances, and essential oils
- Fine and specialty chemicals
- Oil sands: extract bitumen away from water

Why LLE is used?

- Temperature sensitive products
- High purity requirements
- High-boiling point species in low quantity
- Need to separate by species type (rather than relative volatility)
- Close-boiling points, but high solubility difference
- Azeotrope – forming mixtures



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Extraction process	feed	solvent
Separation of aromatic hydrocarbons from petroleum fractions	Petroleum fractions containing aromatics	Tetraethylene glycol- water
Extraction of phenolics from coal tar and liquors	Light liquor containing phenolics (obtained in a coal carbonization plant)	Isobutyl acetate
Extraction of phenol from aqueous wastes	Aqueous waste streams, coal carbonization plants. Phenolic resin plants	Butyl acetate, di- isopropyl ether, MIBK
Extraction of citral (a flavouring substance used in food and some other industries)	Lemon grass and orange oil	An alcohol and a lower hydrocarbon A polar solvent like acetone or a lower alcohol
Removal of H ₂ S	Liquified natural gas (LNG)	Monoethanolamine (MEA), Methanol- diethanolamine (MDEA)



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- Caprolactum is produced and used in large quantities for the manufacture of nylon-6, a bulk polymer. It is synthesized from cyclohexane (cyclohexane → cyclohexanol → cyclohexanone → cyclohexanone oxime → caprolactum)
- The last step of the process involves reaction of the oxime with sulphuric acid where caprolactum and ammonium sulphate are formed.
- The aqueous reaction mixture separates into an organic-rich phase (lactum oil contains 65-70%) and a water-rich phase having ammonium sulphate and 2-3% caprolactum.
- The two phases are fed at appropriate locations of a rotating disk contactor. Toluene and, benzene and trichloroethylene are good solvents (the last two are not used any more)
- Caprolactum goes to the extract and ammonium sulphate remains in the raffinate. The two streams are further treated to get caprolactum in a pure form and to recover the solvent and ammonium sulphate



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Operating Modes of Extraction

- Batch or continuous extractions
- Batch extraction – single stage or multiple stage
- Continuous extraction – co-current or countercurrent extraction
- Multiple stage is necessary to achieve high bioproduct recovery in extraction



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Batch Extraction

- The aqueous feed is mixed with the organic solvent
- After equilibrium, the extract phase containing the desired solute is separated out for further processing.
- A schematic representation of a single batch operation.

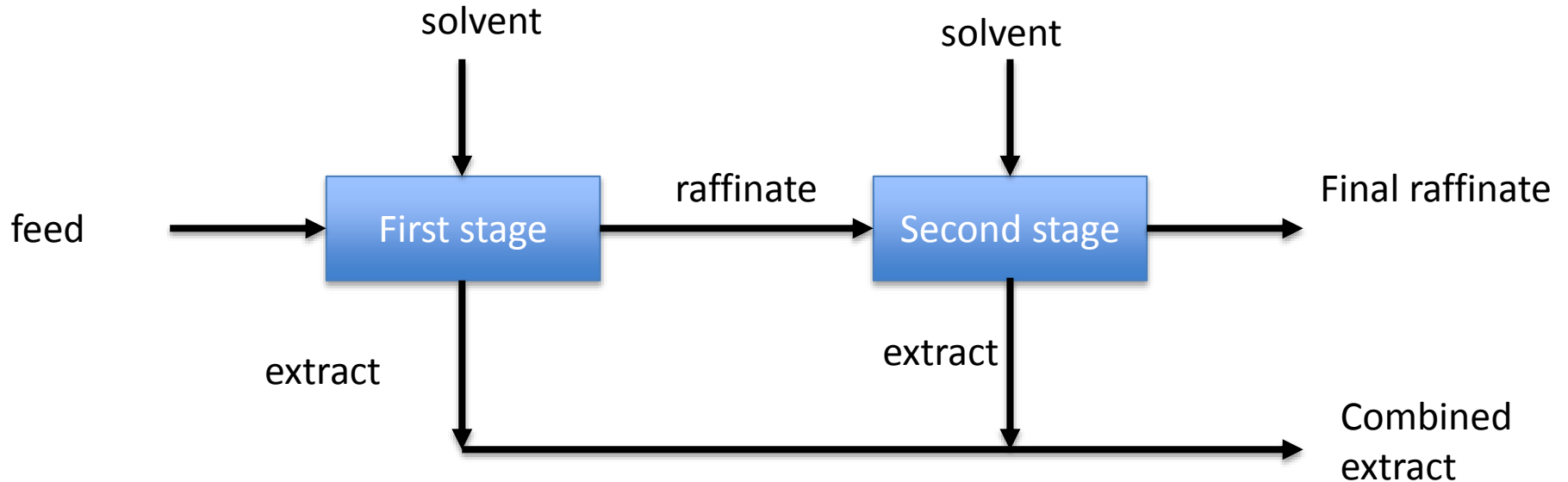


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Batch Extraction in Multiple Stage

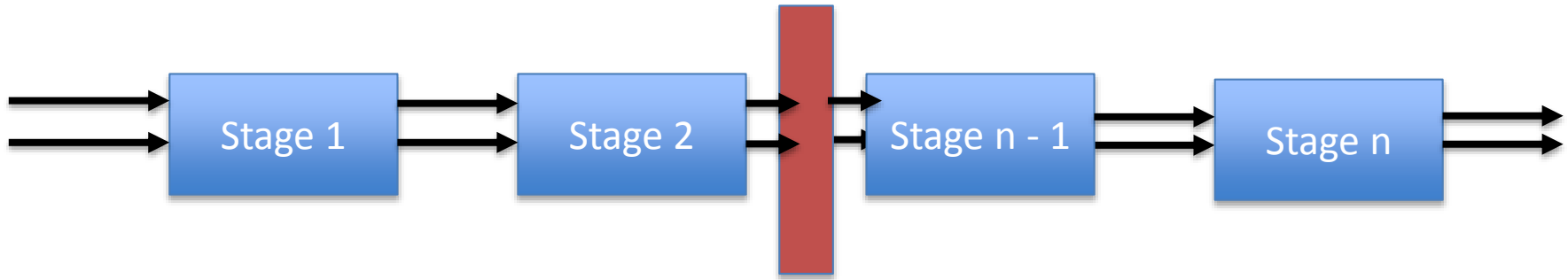


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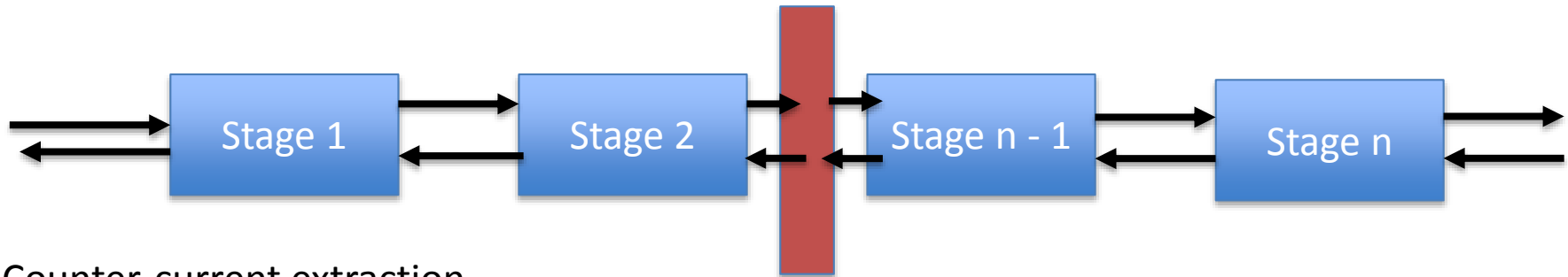
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Continuous Extraction



Co-current extraction



Counter-current extraction

Counter-current flow is preferable because the solute concentration gradient (driving force) is larger



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Extraction of dilute solution

- Extraction factor:
- $E = \frac{K_D V}{L}$
- Where
- E = extraction factor
- K_D = distribution coefficient
- V = volume of solvent
- L = volume of aqueous



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Extraction of Dilute Solution

- For a single-stage extraction with pure solvent:
- - the fraction of solute remaining is:
- $\frac{1}{1+E}$
- The fraction recovered is:
- $\frac{E}{1+E}$



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Extraction of Concentrated Solution

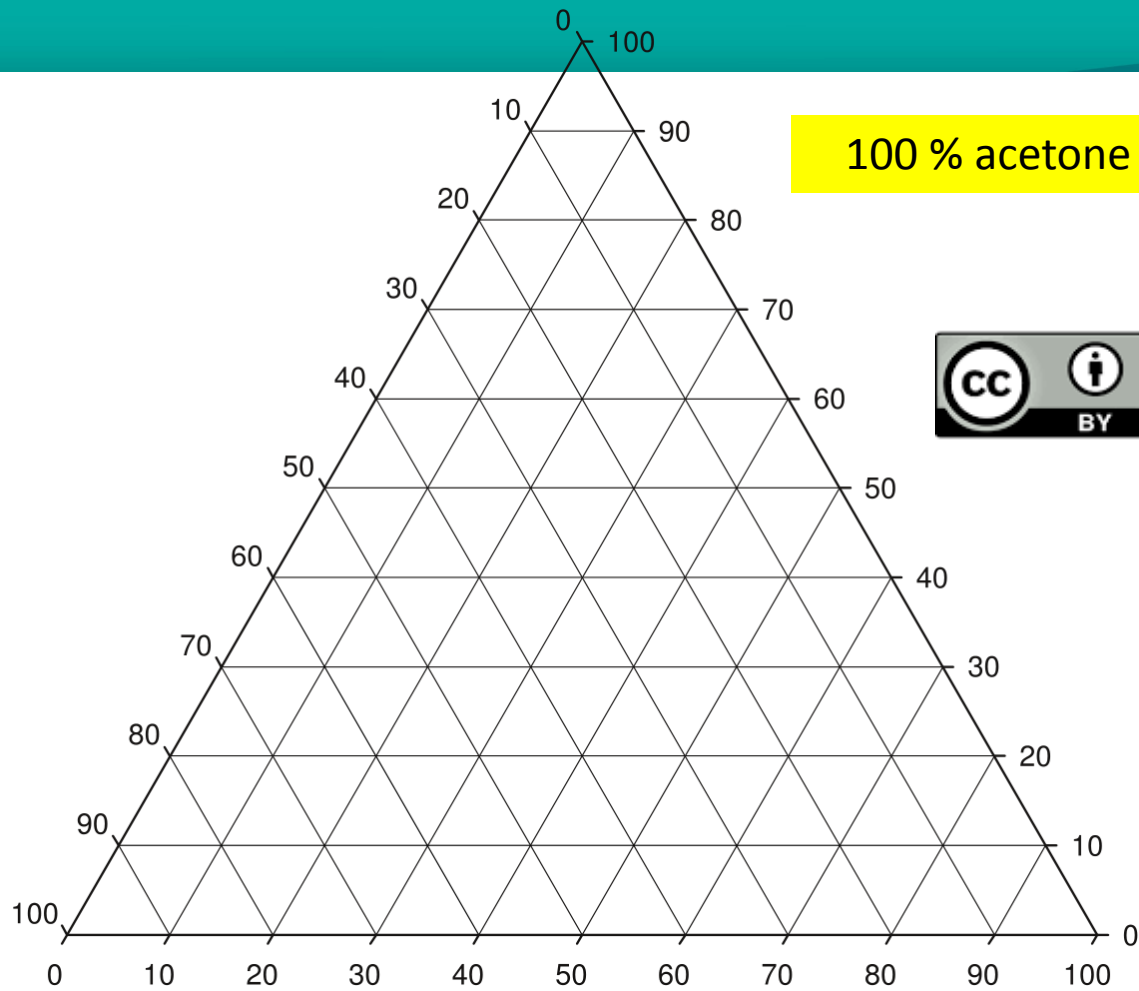
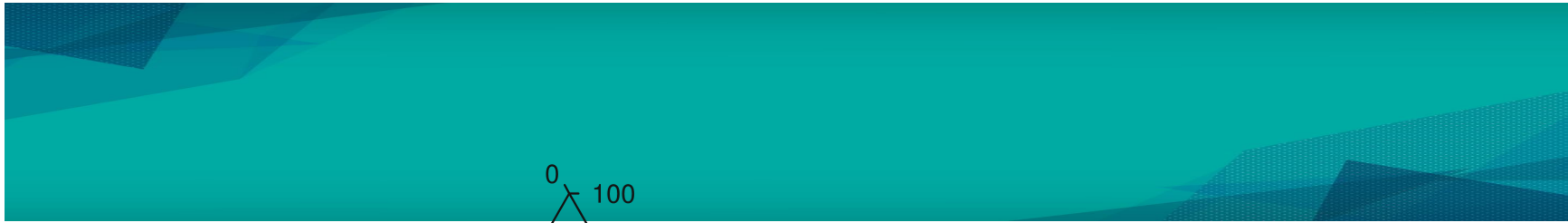
- Equilibrium relationships are more complicated
 - 3 or more components present in each phase
- Equilibrium data are often presented on a triangular diagram



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100 % acetone

100 % MIBK

100 % water



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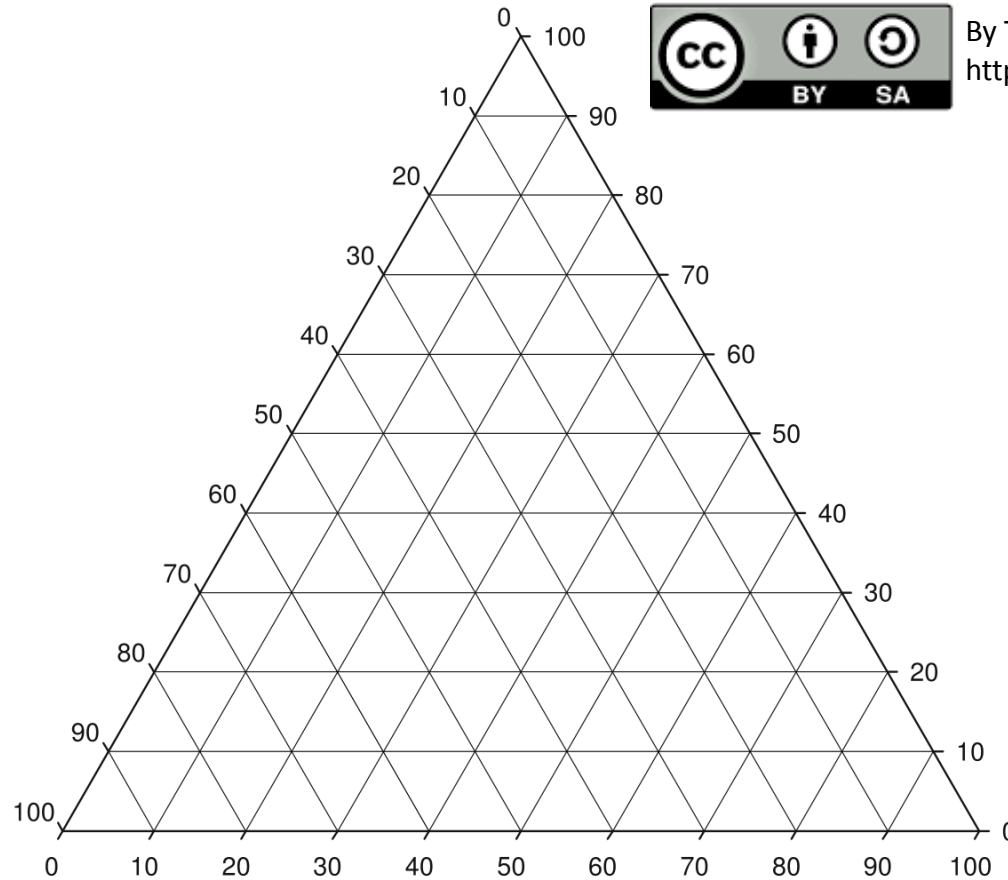
Single Phase Liquid



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Solution A
70% acetone
10% water
20% MIBK

Solution B
33% acetone
33% water
33% MIBK



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Conclusion of The Chapter

- This chapter discussed about solvent extraction, operating modes of extraction, batch and continuous extraction, extraction of dilute and concentrated solution, choice of solvent, and extraction equipment



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