

Separation Processes, ChE 4M3, 2012

Assignment 5

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Objectives: Confirming your understanding of liquid-liquid extraction and adsorption.

Question 1 [20]

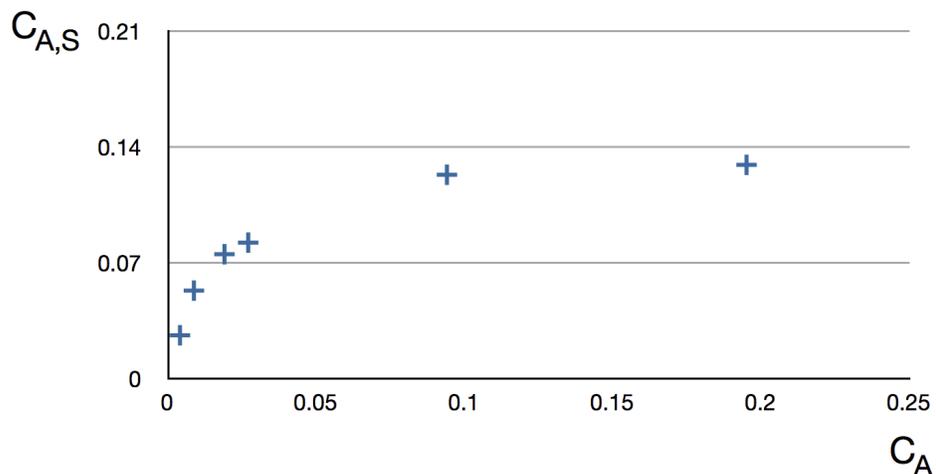
2.5 m³ of wastewater solution with 0.25 kg phenol.m⁻³ is mixed in a batch reactor with 3.0 kg granular activated carbon until equilibrium is reached. Use the following values determined in the lab, calculate the final equilibrium values of phenol extracted and percent recovery.

C_A	$C_{A,S}$
0.004	0.026
0.0087	0.053
0.019	0.075
0.027	0.082
0.094	0.123
0.195	0.129

1. Plot the isotherm.
2. Determine the isotherm type.
3. Calculate the isotherm parameters.
4. Calculate the steady-state conditions in the batch reactor for C_A in g.cm⁻³ and for $C_{A,S}$ in grams solute per gram of activated carbon.
5. Calculate the percentage amount of phenol that is recovered by the adsorbent.

Solution

1. The isotherm is plotted as:



2. It is a Langmuir isotherm, apparent from the plot's shape.

3. Parameters for the isotherm are estimated so that $C_{A,S} = \frac{K_5 C_A}{K_6 + C_A} = \frac{0.140 C_A}{0.0162 + C_A}$. See [this spreadsheet for the detailed calculations](#). You must use an [Eadie-Hofstee plot](#) where you plot $y = C_{A,S}$ vs $x = \frac{C_{A,S}}{C_A}$. Then the slope is $-K_6$ and the intercept is K_5 . The [Lineweaver-Burk plot](#) accumulates too much error and should not be used. Also, it is not valid to use 2 of the data points to back-calculate 2 unknowns: if they were measured in error, then those parameters will be poorly estimated.
4. There are 625 g of phenol added to the batch system. This separates into two components: the amount adsorbed and the amount remaining in solution:

$$0.625 = (2.5 \text{ m}^3)(C_A) + (3 \text{ kg})(C_{A,S})$$

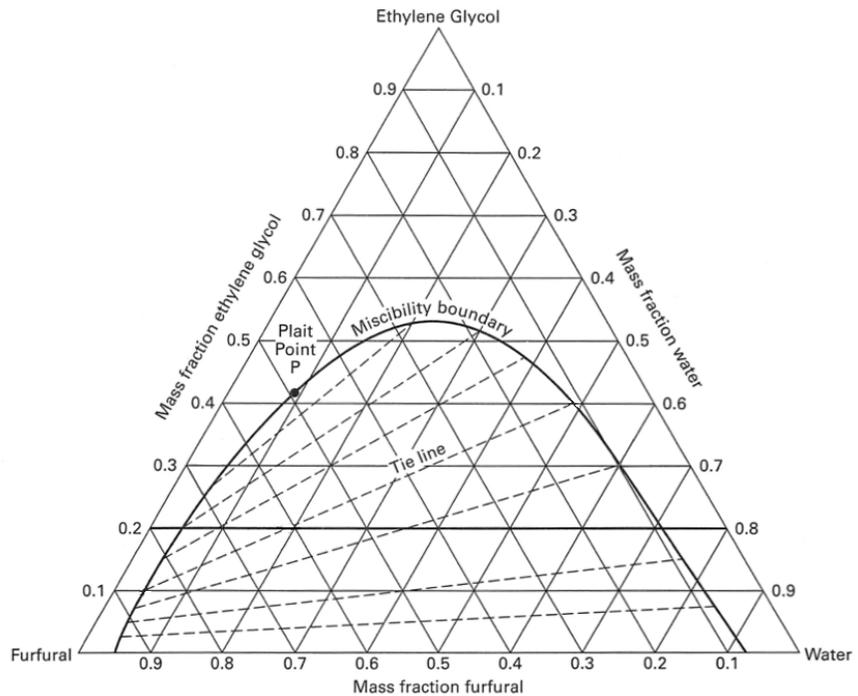
After adjusting the above equation for units, we see it is a straight line that can be plotted over the isotherm. It passes through the point $(C_A, C_{A,S}) = (0, 0.208)$ and $(C_A, C_{A,S}) = (0.25, 0)$ on the x - and y -axes respectively. It is left up to you to connect these points on the plot. You will find where it crosses the isotherm, which is approximately at $C_A = 0.1 \text{ g}\cdot\text{cm}^{-3}$. This is the final equilibrium point. The concentration on the adsorbent at this point is approximately $C_{A,S} = 0.120$ grams solute per gram of activated carbon (from the isotherm equation).

Alternatively you may solve the linear equation above and the non-linear Langmuir isotherm equation for the two unknowns. Or do it graphically as described above.

5. The phenol recovered onto the adsorbent is $0.120 \text{ g/g} \times 3000 \text{ g} = 360 \text{ g}$ of the original 625g added. This is a recovery of about 58%.

Question 2 [20]

The aim is to recover ethylene glycol from water using furfural as solvent (the ternary diagram for furfural, ethylene glycol and water at the unit's operating temperature is provided).



- Calculate the equilibrium compositions leaving a single mixer-settler unit operated at:
 - the recycled solvent stream: $105 \text{ kg}\cdot\text{hr}^{-1}$ with a composition of 84% furfural, and unrecovered ethylene glycol at 5%, with the rest being water.
 - the feed stream contains 42% ethylene glycol and 58% water at a rate of $200 \text{ kg}\cdot\text{hr}^{-1}$.
- Calculate the flow rates of the extract and raffinate streams.
- What is the distribution (partition) coefficient, D_A , for A = ethylene glycol (see course notes for definition)?
- What is the distribution (partition) coefficient, D_C , for C = water, using this same definition?
- What is the separation factor for $i = \text{ethylene glycol}$ from $j = \text{water}$ in the 1=extract and 2=raffinate streams? Also show the relationship between the separation factor and the previous two distribution coefficients.
- What will happen to the separation factor when operating at a lower feed flow rate; show a construction on your diagram to demonstrate it.

Solution

- From the ternary diagram constructions we obtain (approximately):

<i>E</i>	<i>R</i>
$y_{E,A} = 0.09$	$x_{R,A} = 0.37$
$y_{E,S} = 0.87$	$x_{R,S} = 0.10$
$y_{E,C} = 0.04$	$x_{R,C} = 0.53$
$E = 73 \text{ kg}\cdot\text{hr}^{-1}$	$R = 232 \text{ kg}\cdot\text{hr}^{-1}$

- See above, also calculated from the lever rule on the diagram.
- Distribution (partition) coefficient for A = ethylene glycol = $D_A = \frac{y_{E,A}}{x_{R,A}} = \frac{0.09}{0.37} = 0.24$
- Distribution (partition) coefficient for C = water = $D_C = \frac{y_{E,C}}{x_{R,C}} = \frac{0.04}{0.53} = 0.075$
- The separation factor is $S_{i,j} = \frac{x_{i,1}/x_{j,1}}{x_{i,2}/x_{j,2}} = S_{A,C} = \frac{x_{E,A}/x_{E,C}}{x_{R,A}/x_{R,C}} = \frac{x_{E,A}}{x_{R,A}} \cdot \frac{x_{R,C}}{x_{E,C}} = \frac{D_A}{D_C} = \frac{0.24}{0.075} = 3.2$, which is very low, as expected from the construction on the ternary diagram.
- At a lower feed flow our operating point moves to M^* . From the lever rule you can easily show this implies SM^* length has decreased from SM and length FM^* has increased relative to FM . A tie line through this new operating point leads to equilibrium concentrations:

E^*	R^*
$y_{E^*,A} = 0.06$	$x_{R^*,A} = 0.25$
$y_{E^*,S} = 0.89$	$x_{R^*,S} = 0.09$
$y_{E^*,C} = 0.05$	$x_{R^*,C} = 0.66$

from which we find the new separation factor is $S_{i,j} = S_{A,C} = \frac{x_{E^*,A}/x_{E^*,C}}{x_{R^*,A}/x_{R^*,C}} = \frac{(0.06)/(0.05)}{(0.25)/(0.66)} = 3.16$, which really didn't change our separation factor much. This makes sense from the attached ternary diagram. We see that both the extract and raffinate concentrations dropped. This system has tie lines that don't work in our favour. All we can do is add many sequential stages to the system in order to achieve a higher degree of separation.

Name: _____

$[S] = 105 \text{ kg}$

$x_S = 0.84$

$x_C = 0.11$

$x_A = 0.05$

$\frac{x}{154} = \frac{F_M}{F_S} = \frac{S}{M} = \frac{105}{1057200} \Rightarrow x = 53 \text{ mm}$

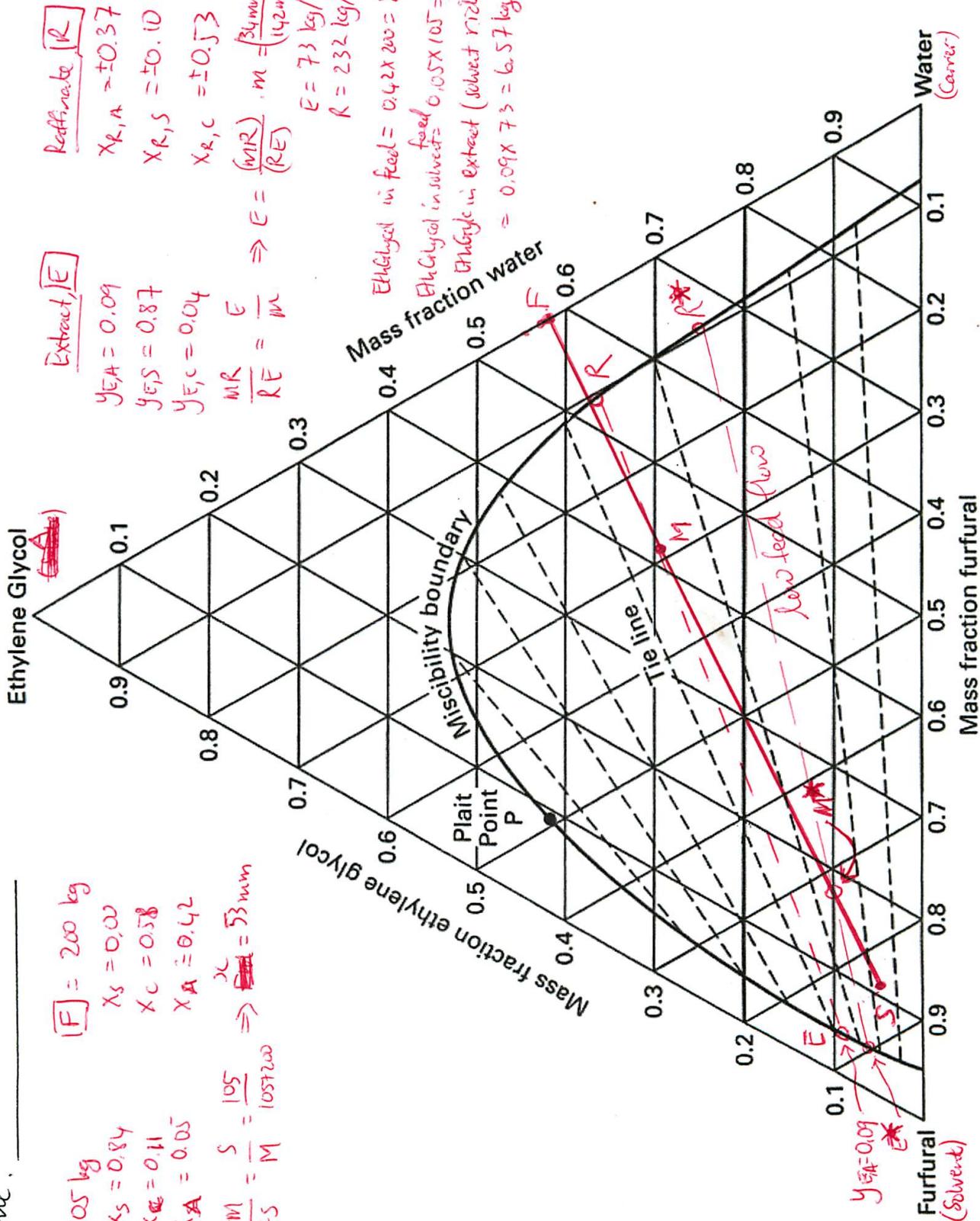
$[F] = 200 \text{ kg}$

$x_S = 0.00$

$x_C = 0.58$

$x_A = 0.42$

$\frac{x}{154} = \frac{F_M}{F_S} = \frac{S}{M} = \frac{105}{1057200} \Rightarrow x = 53 \text{ mm}$



Extract [E]

$y_{EA} = 0.09$

$y_{ES} = 0.87$

$y_{EC} = 0.04$

$\frac{MR}{RE} = \frac{E}{M} \Rightarrow E = \frac{(MR)}{(RE)} \cdot m = \left(\frac{34 \text{ mm}}{142 \text{ mm}}\right) 305$

$E = 73 \text{ kg/hr}$

$R = 232 \text{ kg/hr}$

Residue [R]

$x_{R,A} = \pm 0.37$

$x_{R,S} = \pm 0.10$

$x_{R,C} = \pm 0.53$

$E = 73 \text{ kg/hr}$

$R = 232 \text{ kg/hr}$

Ethylene glycol in feed = $0.4 \times 200 = 84 \text{ kg/hr}$
 Ethylene glycol in solvent = $0.105 \times 105 = 5.25 \text{ kg/hr}$
 Ethylene glycol in extract (subject to 172h) = $0.09 \times 73 = 6.57 \text{ kg/hr}$

Question 3 [40]

Consider a system for which you have been given the ternary diagram. A = solute, S = solvent, C = carrier. The feed, F , enters at 112 kg/hr with composition of 25 wt% solute and 75 wt% carrier, shown as point F .

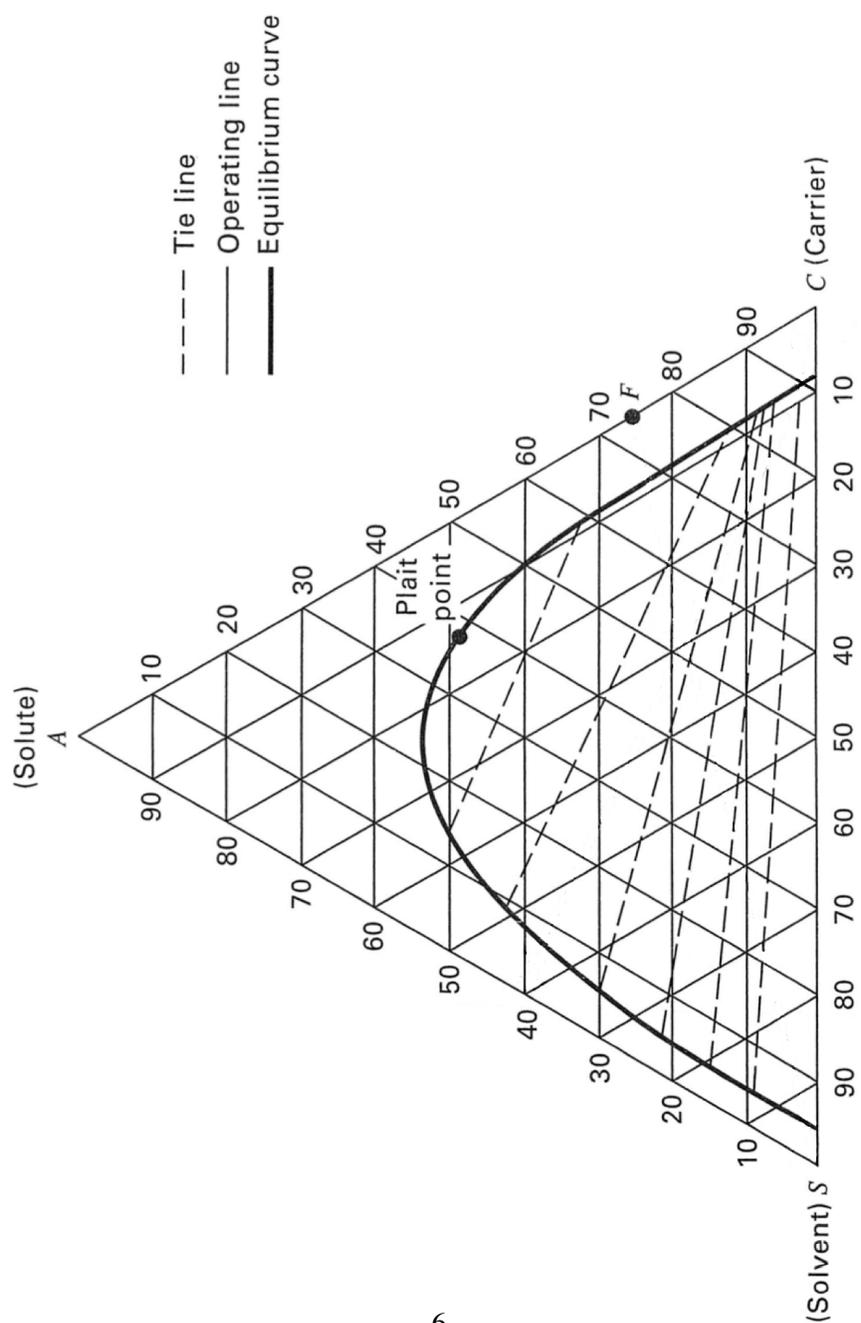
1. Calculate the flow and composition of the extract and raffinate from (*covered in class*):
 - (a) 1st co-current stage, using a pure solvent flow of 50 kg/hr.
 - (b) 2nd co-current stage, which is fed with a fresh solvent flow of 50 kg/hr.
2. For the overall 2-stage system, find the (*covered in class*):
 - (a) overall recovery, defined using the mass flows of $\frac{\text{Feed} - R_2}{\text{Feed}}$
 - (b) overall concentration [kg/kg] of combined extract streams, E_1 and E_2 combined concentration.
3. *New*: The objective now is to have a counter-current system so the raffinate leaving in the N^{th} stage, R_N has $y_{R_N} = 0.025$.
 - (a) What is the *maximum allowable solvent flow* to achieve $y_{R_N} = 0.025$?
 - (b) Explain whether it's possible to achieve a final extract stream of concentration $y_{E_1} = 0.20$.
 - (c) Show the construction on the ternary diagram for the number of equilibrium stages to achieve $y_{R_N} = 0.025$, given a solvent flow of 27.5 kg/hr.
 - (d) Plot, on the same axes, the concentrations in the extract and raffinate streams from each stage.
 - (e) Calculate the distribution (partition) coefficient for the solute in each stage, $D_{A,i} = \frac{y_{E_i,A}}{x_{R_i,A}}$

Submit the single ternary diagram for part (1 and 2), and another ternary diagram for part (3).

Be sure to make a copy of your filled in diagrams, because you might not be able to pick up your graded assignment 5 before the final exam.

Solution

1. These are the flows and compositions from construction on the diagram (lever rule). Note that all flows and compositions are approximate, depending on your plots.
 - (a) $E_1 = 66.3$ kg/hr ($x_{E_1,S} = 0.65$; $x_{E_1,C} = 0.05$; $x_{E_1,A} = 0.30$).
 $R_1 = 95.7$ kg/hr ($x_{R_1,S} = 0.08$; $x_{R_1,C} = 0.83$; $x_{R_1,A} = 0.09$).
 - (b) $M_2 = 58.6$ kg/hr ($x_{M_2,S} = 0.40$; $x_{M_2,C} = 0.54$; $x_{M_2,A} = 0.06$).
 $E_2 = 60.2$ kg/hr ($x_{E_2,S} = 0.85$; $x_{E_2,C} = 0.04$; $x_{E_2,A} = 0.11$).
 $R_2 = 85.6$ kg/hr ($x_{R_2,S} = 0.09$; $x_{R_2,C} = 0.89$; $x_{R_2,A} = 0.02$).
2. For the overall 2-stage system
 - (a) Recovery = $\frac{\text{Feed} - R_2}{\text{Feed}} = \frac{28 - 1.97}{28} \approx 93\%$
 - (b) Combined concentration = $\frac{(E_1)(y_{E_1,A}) + (E_2)(y_{E_2,A})}{E_1 + E_2} = \frac{(66.3)(0.3) + (60.2)(0.11)}{66.3 + 60.2} \approx 0.21$ kg/kg.



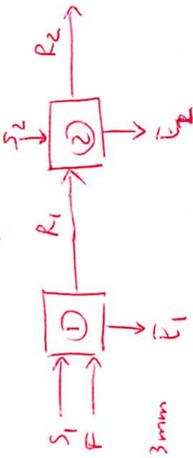
- Tie line
- _____ Operating line
- Equilibrium curve

(Series co-current) = (Cross-current)

$$S_1 F = 99 \text{ mm}$$

$$\frac{M_1 F x_1}{S_1 F} = \frac{x}{99 \text{ mm}} = \frac{S_1}{M_1} = \frac{50}{112+50} \Rightarrow x = 30.5 \text{ mm}$$

$$\frac{M_2 R_1}{S_2 R_1} = \frac{x}{97 \text{ mm}} = \frac{S_2}{M_2} = \frac{50}{95.8+50} \Rightarrow x = 33.3 \text{ mm}$$



Mass flows for E_1 and R_1 are found by lever-rule, or by a mass balance across unit I

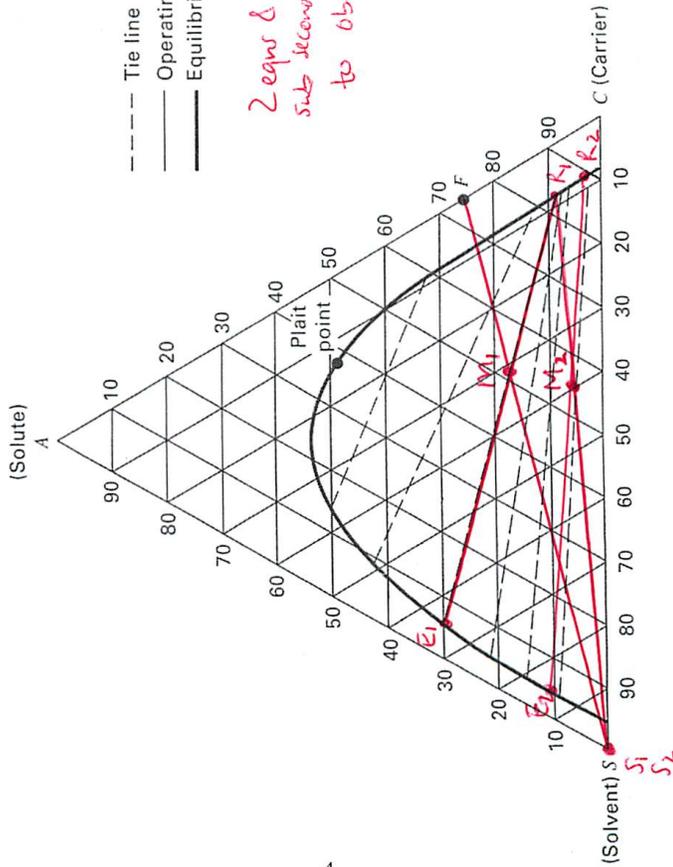
Similarly for E_2 and R_2 flowrates.

e.g. $E_1 + R_1 = M_1 = F + S_1 = 162$

$$y_{E_1} E_1 + x_{R_1} R_1 = x_{M_1} M_1$$

$$\begin{cases} (0.30)(E_1) + (0.08)(R_1) = (0.17)(162) \\ E_1 + R_1 = 162 \end{cases} \Rightarrow \begin{matrix} E_1 = 66.3 \text{ kg/hr} \\ R_1 = 95.7 \text{ kg/hr} \end{matrix}$$

- Tie line
- Operating line
- Equilibrium curve



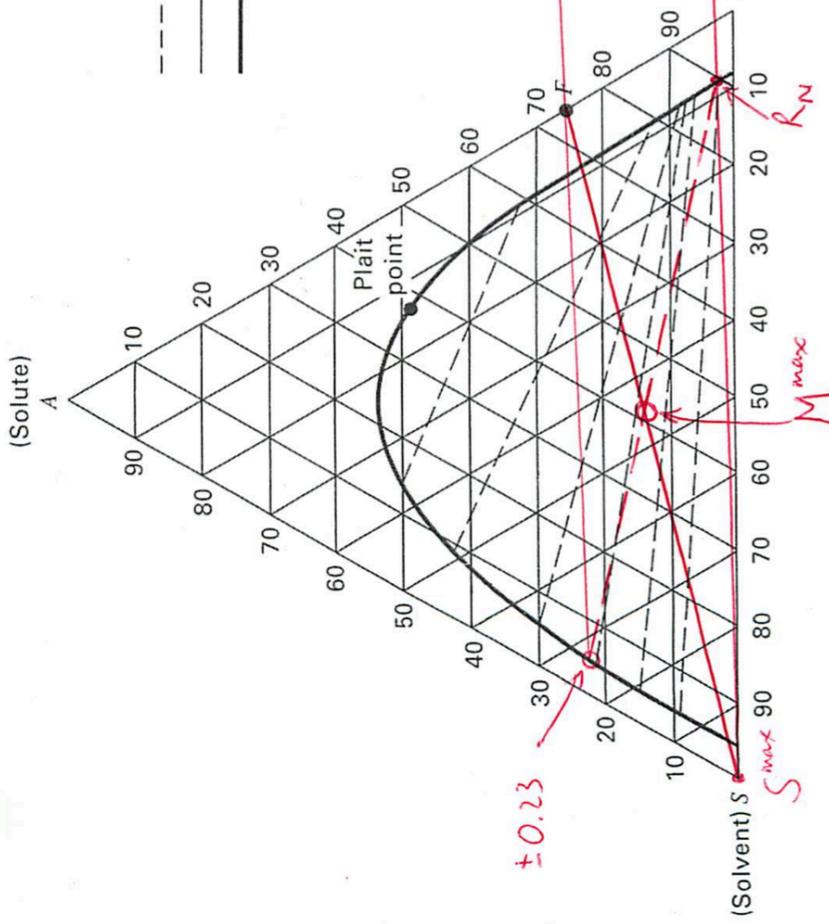
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3. For the counter-current system:

- (a) Draw the operating line from the solvent to the point $y_{R_N} = 0.025$. Draw a parallel line to this, but passing through F . This will find the minimum extract concentration, $y_{E_1} \approx 0.23$. Now directly connect $y_{E_1} \approx 0.23$ to $y_{R_N} = 0.025$: this is the line we would operate along if we essentially had an infinite number of stages. See where this line crosses the line connecting S and F . This is the maximum solvent flow allowed, around $S = 92$ kg (see the illustration below).
- (b) No, as described above, the extract stream must be higher than 23%. Also see the construction on slides 50 and 51 in the class notes.
- (c) The concentrations read from the illustration (below):

E_1	R_1
$y_{E1,A} = 0.49$	$x_{R1,A} = 0.28$
$y_{E1,S} = 0.39$	$x_{R1,S} = 0.09$
$y_{E1,C} = 0.12$	$x_{R1,C} = 0.63$
$D_{A,1} = 0.49/0.28 = 1.75$	
E_2	R_2
$y_{E2,A} = 0.46$	$x_{R2,A} = 0.21$
$y_{E2,S} = 0.43$	$x_{R2,S} = 0.09$
$y_{E2,C} = 0.11$	$x_{R2,C} = 0.70$
$D_{A,2} = 0.46/0.21 = 2.19$	
E_3	R_3
$y_{E3,A} = 0.40$	$x_{R3,A} = 0.11$
$y_{E3,S} = 0.52$	$x_{R3,S} = 0.08$
$y_{E3,C} = 0.08$	$x_{R3,C} = 0.81$
$D_{A,3} = 0.40/0.11 = 3.63$	
E_4	R_4
$y_{E4,A} = 0.22$	$x_{R4,A} = 0.07$
$y_{E4,S} = 0.74$	$x_{R4,S} = 0.08$
$y_{E4,C} = 0.04$	$x_{R4,C} = 0.85$
$D_{A,4} = 0.22/0.07 = 3.14$	
E_5	R_5
$y_{E5,A} = 0.15$	$x_{R5,A} = 0.05$
$y_{E5,S} = 0.81$	$x_{R5,S} = 0.08$
$y_{E5,C} = 0.04$	$x_{R5,C} = 0.87$
$D_{A,5} = 0.15/0.05 = 3.00$	
E_6	R_6
$y_{E6,A} = 0.10$	$x_{R6,A} = 0.025$
$y_{E6,S} = 0.86$	$x_{R6,S} = 0.080$
$y_{E6,C} = 0.04$	$x_{R6,C} = 0.895$
$D_{A,6} = 0.10/0.025 = 4.00$	

- (d) The concentration profiles for the solute are shown below:



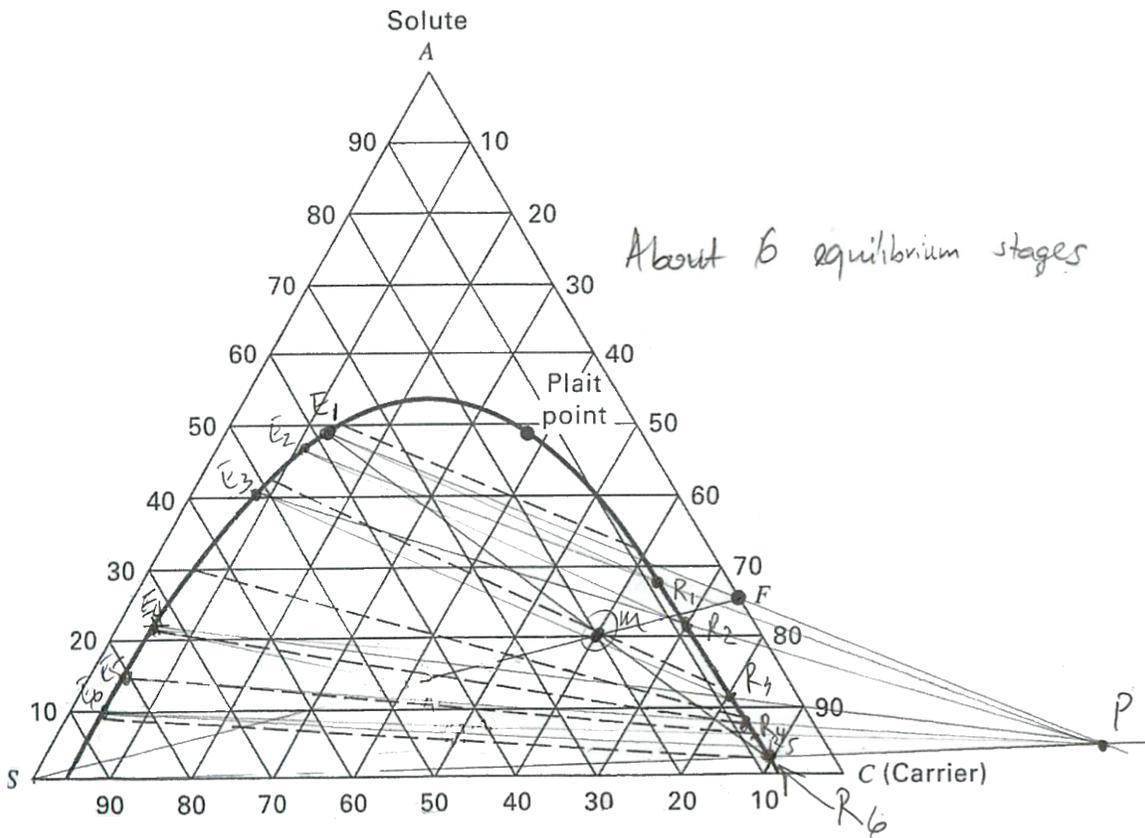
$$\frac{S^{max} M^{max}}{S^{max} F} = \frac{62 \text{ min}}{113 \text{ min}} = \frac{F}{M^{max}} = \frac{F}{F + S^{max}}$$

$F = 112 \text{ kg/hr}$
 $S^{max} \approx 92 \text{ kg/hr}$

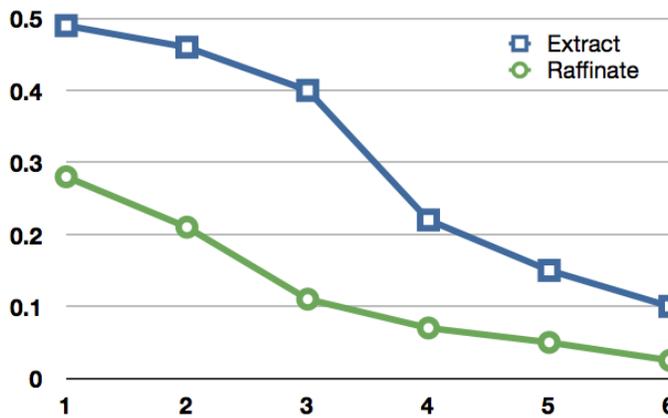
- Tie line
- Operating line
- Equilibrium curve

parallel line to the operating line

operating line



Concentration profile of streams leaving each stage



(e) See the above table for the distribution coefficients.

Question 4 [10]

1. What is the dew point temperature of an air stream containing 15% humidity at 70 °C?
2. What is the amount of water carried in this stream?
3. This stream is cooled adiabatically; what is the adiabatic saturation temperature?
4. What is the amount of moisture held now per kilogram of dry air?

5. *From a previous year's 4M3 assignment:* Read the definition for relative humidity from [The Weather Network](#). Prove that it agrees with our definition in the class.

Solution

1. Answers between 35 and 37 °C are acceptable.
2. Around 0.04 kg water per kg dry air
3. Answers around 41 and 42 °C are acceptable.
4. Answers around 0.051 to 0.055 °C are acceptable.
5. Yes it does: at 20 °C, the humidity of air is saturated at 0.016 kg water per kg dry air, or 16 g/kg. At 50% humidity this drops to 8 grams/kg. The only problem with their definition is that they do not mention the parcel was 1 kg of dry air.