Chapter-4- (الاستخلاص السائل)

liquid-liquid Extraction (solvent extraction)

The separation of the component of a liquid Mixture by treatment with a solvent which one or more of the desired components is preferentially soluble is known as liquid-liquid Extraction. In the operation the liquid-mixture feed and the solvent are partially or completely immiscible.

Three stages are involved:

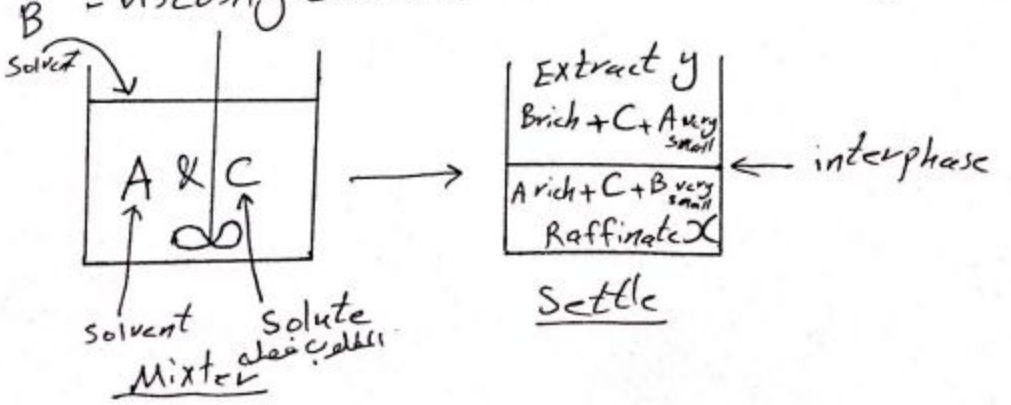
1- Bringing the feed mixture and the solvent into contact.
2- Separation of the two phase.
3- Removal and recovery of the solvent from each phase.

Stage (1) and (2) is carried out in stage wise equipment (single piece of equipment) which is known as Mixer-Settler.

The operation of mixer-settler is:

A- The mixing of the two liquid phase by agitation.
B- The settling in a separate vessel by gravity.

* for example of liquid-liquid Extraction is the separation of aromatic from kerosen to improve their burning qualities.
And the separation of aromatic from paraffin and naphthenic compounds to improve the temperature - viscosity characteristic of lubricating oils.



By weight fraction:

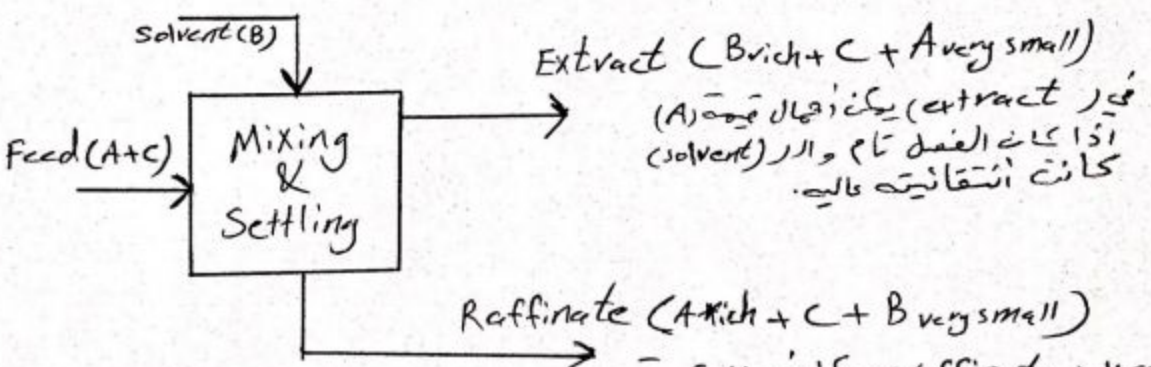
$$y = \frac{C}{B+C+A^{\text{very small}}} = \left(\frac{C}{B+C}\right)_E$$

or $Y = \left(\frac{C}{B}\right)_E$ = mass ratio of solute in extract

$$x = \frac{C}{A+C+B^{\text{very small}}} = \left(\frac{C}{A+C}\right)_R$$

$X = \left(\frac{C}{A}\right)_R$ = mass ratio of solute in raffinate

عندنا مادتين ممتزجة (miscible) وهي (A+C) نضيف مادة B (solvent) بحيث تكون لها القابلية على سحب المادة C الملوث فصلها من A (has good selectivity).



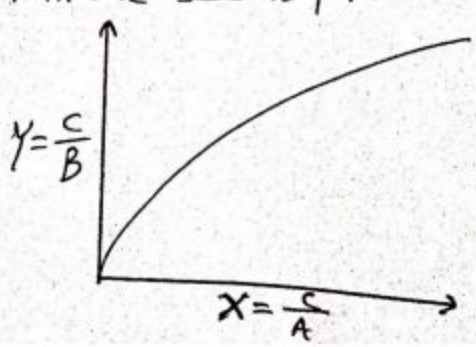
في الـ (extract) يمكن إهمال قيمة (A) إذا كان الفصل تام والـ (solvent) كانت انتقائيته عالية.

في الـ (raffinate) يمكن إهمال قيمة (B) إذا كان الفصل تام وغير ممتزج.

The addition of a new solvent to a binary mixture of (solute in a solvent) may lead to the formation of several types of mixture.

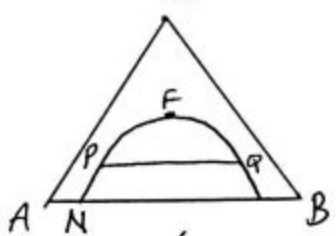
1- The solvent (B) may be completely immiscible with initial solvent (A). (فصل تام وغير ممتزج كلياً)

The equilibrium relation is shown by a plot of the concentration of solute in one phase against the concentration in the second phase.



2- The homogenous solution may be formed, then the selected solvent (B) is unsuitable
تكوين محلول متجانس (غير مفصول الى طبقتين) وفي هذه الحالة يكون المذيب (solvent) المستخدم غير ملائم.

3- The solvent (B) may be partially immiscible with the original solvent resulting in the formation one pair of partially miscible liquid. equilibrium are usually represented by triangular diagrams.
(انفصل جزئي)



Choosing of solvent:

1- Good selectivity (β)

$$\beta = \frac{\text{wt. fraction of C in Extract / wt. fraction of A in Extract}}{\text{wt. fraction of C in Raffinate / wt. fraction of A in Raffinate}}$$

$$\beta = \frac{y_{CE}/y_{AE}}{x_{CR}/x_{AR}}$$

$\beta > 1$

the greater the value of β is the better in the extracting solvent.

2- good recoveribility قابلية استرداد عالية

3- The liquid use may be have low viscosity.

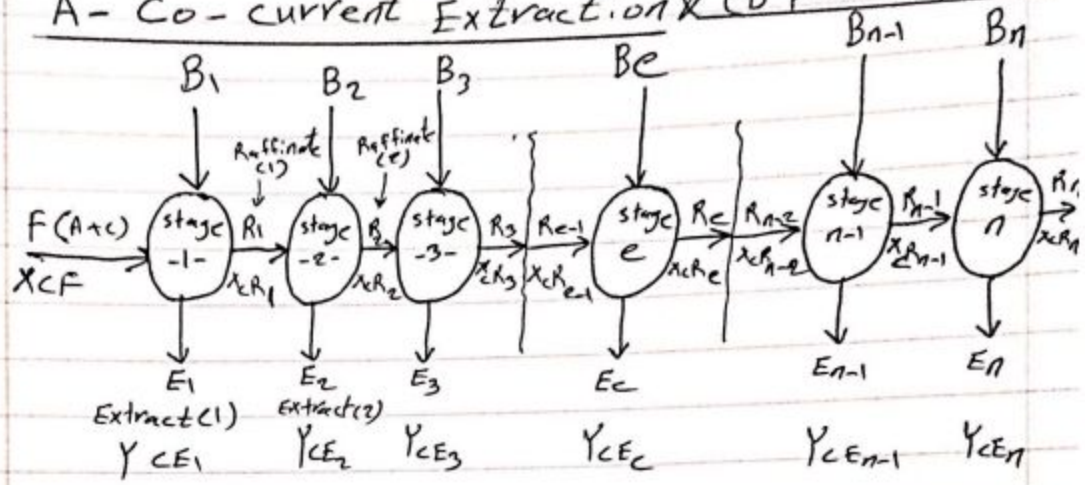
4- Density difference (the greater the density difference. the quicker separater

5- ويجب أن يكون غير سام ورخيص ولا يؤدي الى حرق الجلد عند الاستعمال ولا يسبب التآكل ولا يتجمد عند درجة الحرارة الواطئة.

Extraction (2) عملية

(I) The solvent (B) may be completely immiscible with initial solvent (A). مذيب تام الاختلاط

A- Co-current Extraction & (B pure component)



$$\text{wt. ratio} \quad X = \frac{\text{wt. C}}{\text{wt. A}}$$

$$Y = \frac{\text{wt. C}}{\text{wt. B}}$$

component (c) mass balance on stage (1)

$$0 + A\,X_{CF} = A\,X_{CR_1} + B_1\,Y_{CE_1}$$

$$0 + A \times \frac{C}{A} \quad A\,\frac{C}{A} + B_1 \times \frac{C}{B_1}$$

or $$\boxed{Y_{CE_1} = \frac{-A}{B_1}\,(X_{CR_1} - X_{CF})}$$

component (c) mass balance on stage (2)

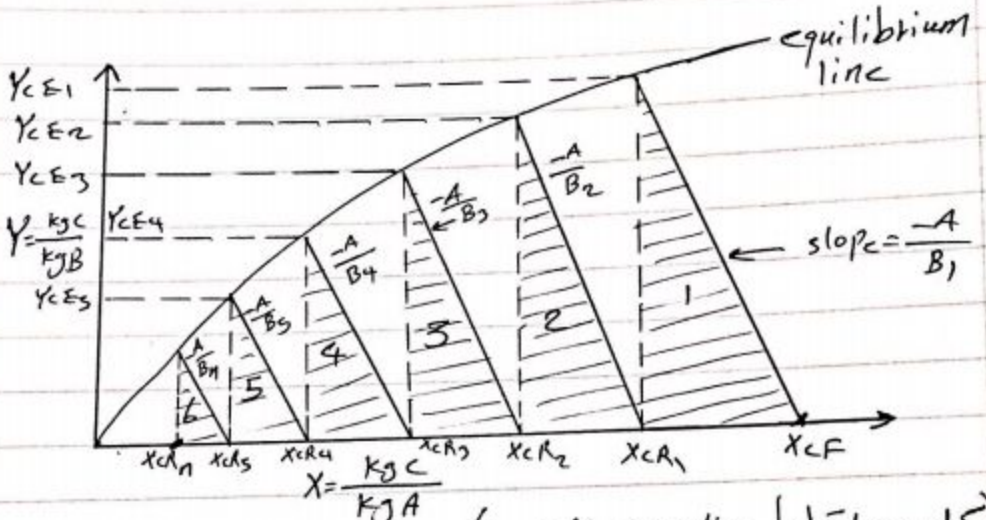
$$0 + A X_{CR_1} = A\,X_{CR_2} + B_2\,Y_{CE_2}$$

or $$\boxed{Y_{CE_2} = \frac{-A}{B_2}\,(X_{CR_2} - X_{CR_1})}$$

(5)

$$\therefore \boxed{Y_{cE_n} = \frac{-A}{B_n}\left(X_{cR_n} - X_{cR_{n-1}}\right)}$$

operating line equation with slope $= \frac{-A}{B_n}$



(لكل مرحلة لها خط operating line)

في العادة نكون هناك عدد (٦) مراحل للمرور من $X_{cF} \rightarrow X_{cR_n}$

B- Co-Current Extraction & B contain some amount of c having composition of Y_{cs}

Material Balance on stage (1)

$$A\,X_{cF} + B_1\,Y_{cs} = A\,X_{cR_1} + B_1\,Y_{cE_2}$$

$$Y_{cE_1} = \frac{-A}{B_1}\left(X_{cR_1} - X_{cF}\right) + Y_{cs}$$

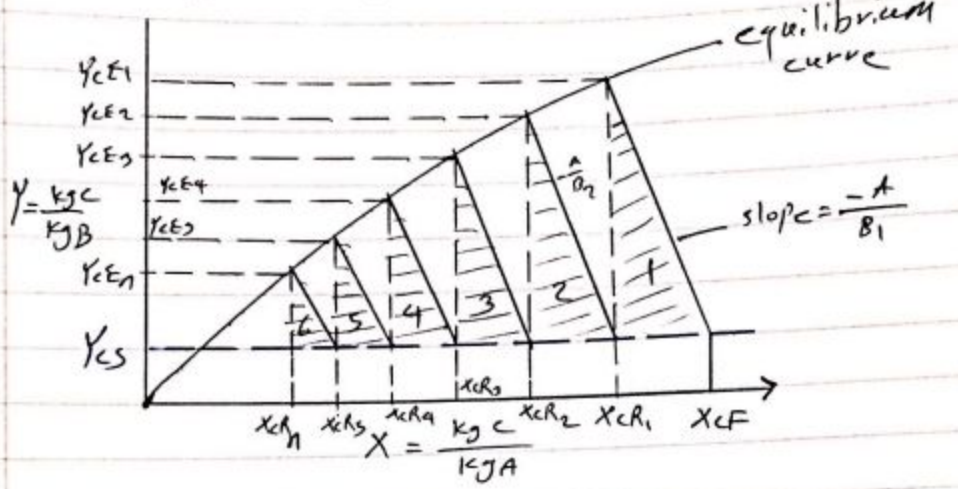
M.B on stage (n)

$$A\,X_{cR_{n-1}} + B_n\,Y_{cs} = A\,X_{cR_n} + B_n\,Y_{cE_n}$$

$$\boxed{Y_{cE_n} = \frac{-A}{B_n}\left(X_{cR_n} - X_{cR_{n-1}}\right) + Y_{cs}}$$

(6)

operating line equation.



If B is added in added in a constant mass in each stage the slope of operating line will be constant, that means all the operating line will be parralal.

C- Co-current etraction and equilibrium data can be represented by a straight line i.e $Y = mX$ and the same amount of pure(B) is added in each stages.

component (c) Material Balance on stage (1)

$$A X_{cF} + 0 = A X_{cR_1} + B Y_{cE_1}$$

$$Y_{cE_1} = m X_{cR_1}$$

$$A X_{cF} = A X_{cR_1} + B m X_{cR_1}$$

$$X_{cR_1} = \left(\frac{A}{A + Bm}\right) X_{cF} \quad \text{——} (1)$$

component (c) Material Balance on stage (2)

$$A X_{cR_1} + 0 = A X_{cR_2} + B Y_{cE_2}$$

$$A X_{cR_1} = A X_{cR_2} + B m X_{cR_2}$$

(7)

$$x_{CR_2} = \left(\frac{A}{A+BM}\right) x_{CR_1} \quad \text{——(2)}$$

$$x_{CR_2} = \left(\frac{A}{A+BM}\right)^2 x_{CF} \quad \text{——(3)}$$

$$x_{CR_n} = \left(\frac{A}{A+BM}\right)^n x_{CF} \quad \text{——(4)}$$

The number of stage required to go from x_{CF} to $x_{CR_n} = n$

$$\frac{x_{CR_n}}{x_{CF}} = \left(\frac{A}{A+BM}\right)^n$$

$$\ln \frac{x_{CR_n}}{x_{CF}} = n \ln \left(\frac{A}{A+BM}\right)$$

$$\boxed{n = \frac{\ln \frac{x_{CR_n}}{x_{CF}}}{\ln \frac{A}{A+BM}}}$$

n = Number of stage required.

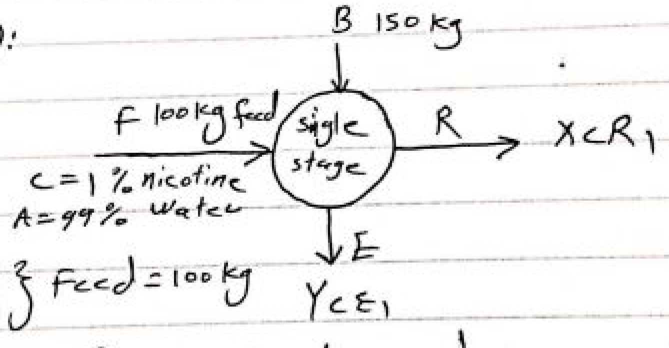
محاضرة (3)

Example (1): Nicotine (c) in a water (A) solution containing 1% nicotine is to be extracted with kerosen (B) at 20°c. Water & kerosen are essentially insoluble. a) Determine the percentage extraction of nicotine if 100 kg of feed solution is extracted once with 150 kg solvent (one stage).
b) Repeat for three continuous theoretical stage extractions using 50 kg solvent in each stage.

Equilibrium data are:

$X \; \frac{\text{kg nicotine}}{\text{kg water}}$	0	0.001	0.0024	0.005	0.0075	0.0099	0.02
$Y \; \frac{\text{kg nicotine}}{\text{kg kerosine}}$	0	0.00081	0.0019	0.0045	0.0068	0.0091	0.018

Solution (a):



C = 1 kg, A = 99 kg } Feed = 100 kg

$$X_{CF} = \frac{C}{A} \text{ in feed} = \frac{1}{99} = 0.0101$$

$$\frac{-A}{B} = \frac{-99}{150} = -0.66 \text{ slope of operating line}$$

From X_{CF} in drawing of X & Y draw line FD with a slope of -0.66 and Find:

$$X_{CR1} = 0.00425 \; \frac{\text{kg nicotine}}{\text{kg water}} = \frac{C}{A}$$

$$Y_{CE1} = 0.0038 \; \frac{\text{kg nicotine}}{\text{kg kerosine}} = \frac{C}{B}$$

⑦

The nicotine removed from water = $\left(0.0101 \frac{\text{kg nicotine}}{\text{kg water}} - \frac{0.004 \text{ kg nicotine}}{\text{kg water}}\right)$ × 99 kg water

= 0.58 kg

= 58% percent extraction of nicotine

b) For each stage, the slope of operating line

$= -\frac{A}{B} = \frac{50}{99} = -1.98$.

from X_{CF} in the drawing, draw three stage with slope = −1.98. we find that the final raffinate composition is $X_{CR_3} = 0.0034$

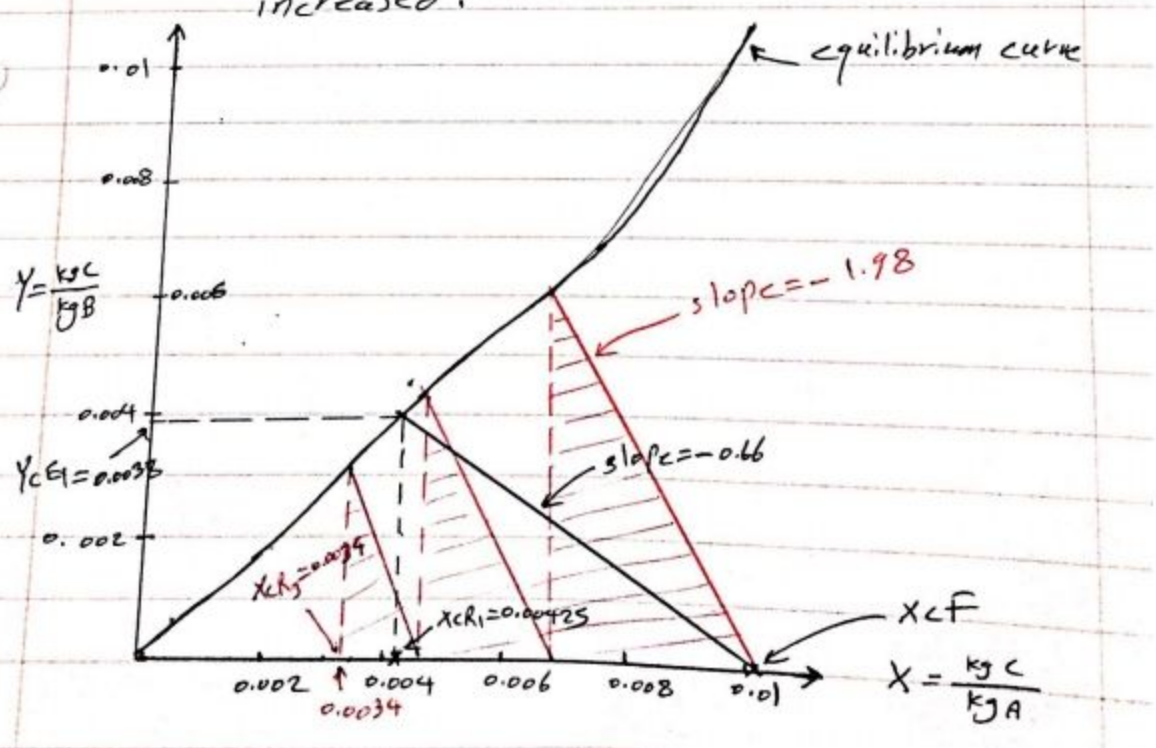
∴ The nicotine removed from water

$= \left(0.0101 \frac{\text{kg nicotine}}{\text{kg water}} - 0.0034 \frac{\text{kg nicotine}}{\text{kg water}}\right) \times 99$ kg water

= 0.663 kg

= 66.3% of that in the feed

Notice: when we use three stages the % removed increased.

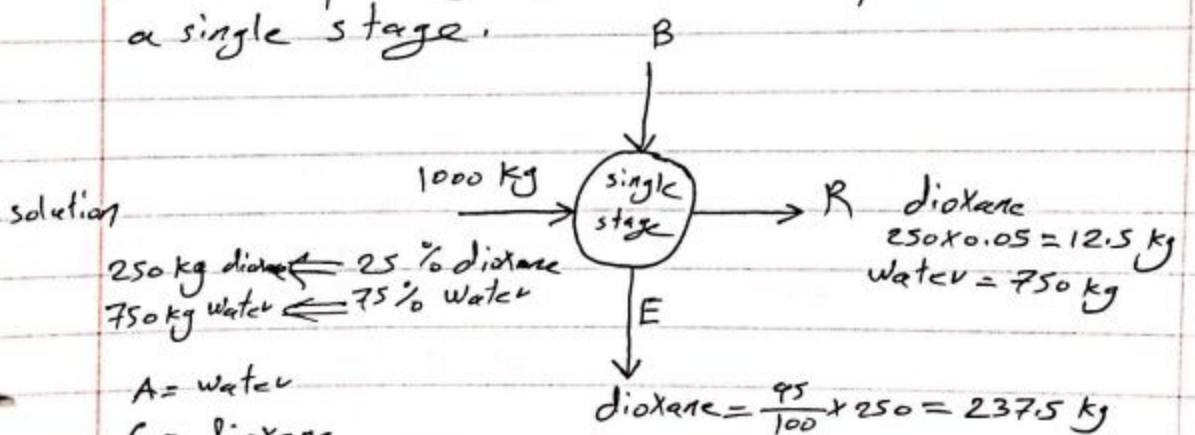


(10)

Example: Water dioxane solution for a minimum boiling Azeotrope at atm. pressure and can not be separated by ordinary distillation method. Benzene forms no Azeotrope with dioxane and may be used as an Extracting solving at 25°c. The equilibrium distribution of dioxane between water & benzene is as follows.

wt.% dioxane in water (x)	0.051	0.189	0.252
wt% dioxane in Benzene (y)	0.052	0.225	0.32

The feed is 1000 kg of 25% dioxane & 75% water and Extraction design to remove 95% of dioxane. Find the quantity of solvent (B) required in a single stage.



A = water
C = dioxane
B = Benzene

$$y = \frac{C}{C+B} = \frac{\frac{C}{B}}{\frac{C}{B}+\frac{B}{B}} = \frac{Y}{Y+1}$$

$$\therefore Y = \frac{y}{1-y}$$

$$x = \frac{C}{C+A} = \frac{\frac{C}{A}}{\frac{C}{A}+\frac{A}{A}} = \frac{X}{X+1}$$

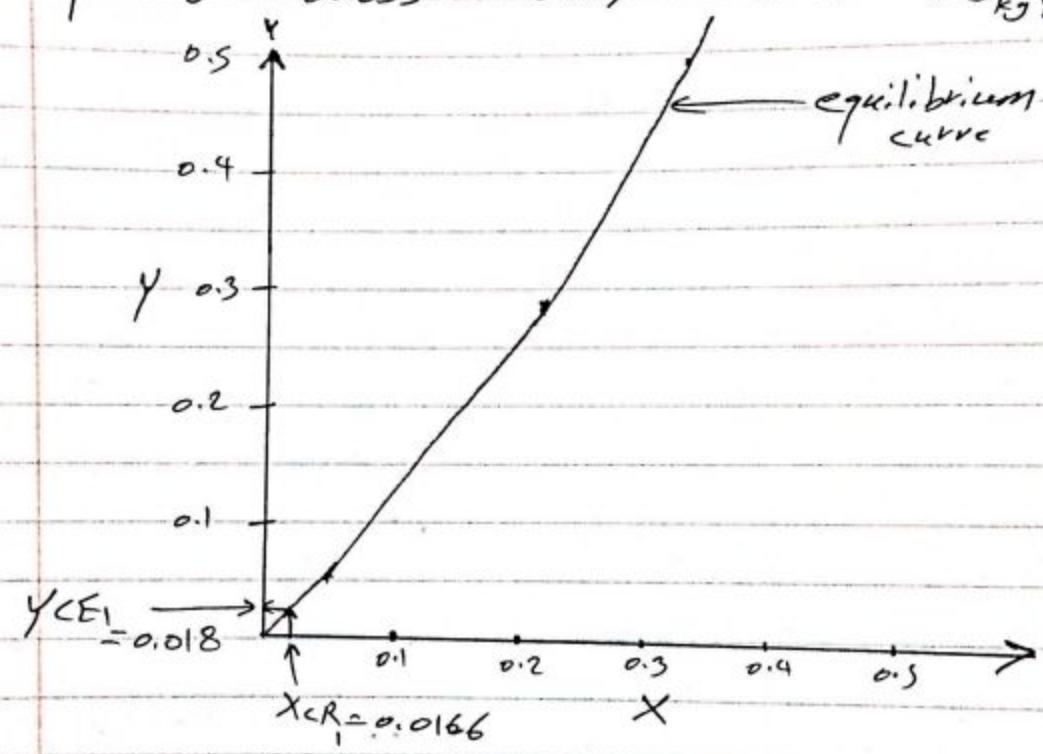
$$X = \frac{x}{1-x}$$

(11)

$$X_{CR} = \frac{C}{A} = \frac{12.5 \text{ kg dioxane}}{750 \text{ kg water}} = 0.0166$$

Mass Ratio إلى Mass fraction عن طريق equilibrium يتم تحويل الـ

X	0	0.0537	0.233	0.337	$\frac{\text{kg dioxane}}{\text{kg water}}$
Y	0	0.055	0.29	0.47	$\frac{\text{kg dioxane}}{\text{kg benzene}}$



From Diagram above

$$X_{CR_1} = 0.0166 \Longrightarrow Y_{CE_1} = 0.018 \ \frac{\text{kg dioxane}}{\text{kg benzene}}$$

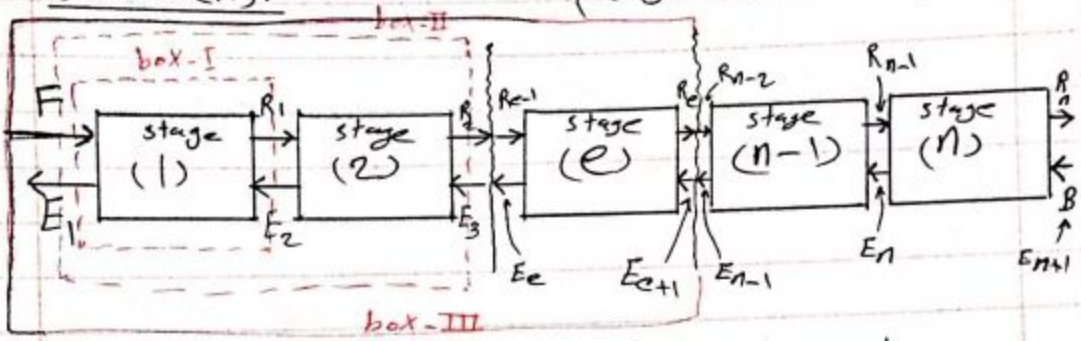
$$0.018 = \frac{237.5 \text{ kg dioxane}}{B}$$

$$\therefore B = \frac{237.5}{0.018} = 13194 \text{ kg}$$

D- counter current extraction :

Solvent (B) may be completely immiscible with intial solvent (A).

الاتجاه المعاكس وتفصل تام



All Raffinate stream (R) contain $\frac{C}{A}$ only

All Extract stream (E) contain $\frac{C}{B}$ only

component C material balance on 1st stage box-I

$$AX_{CF} + BY_{CE_2} = AX_{CR_1} + BY_{CE_1}$$

$$\boxed{Y_{CE_2} = \frac{A}{B}(X_{CR_1} - X_{CF}) + Y_{CE_1}}$$

Material Balance on 2nd stage box-II

$$AX_{CF} + BY_{CE_3} = AX_{CR_2} + BY_{CE_1}$$

$$\boxed{Y_{CE_3} = \frac{A}{B}(X_{CR_2} - X_{CF}) + Y_{CE_1}}$$

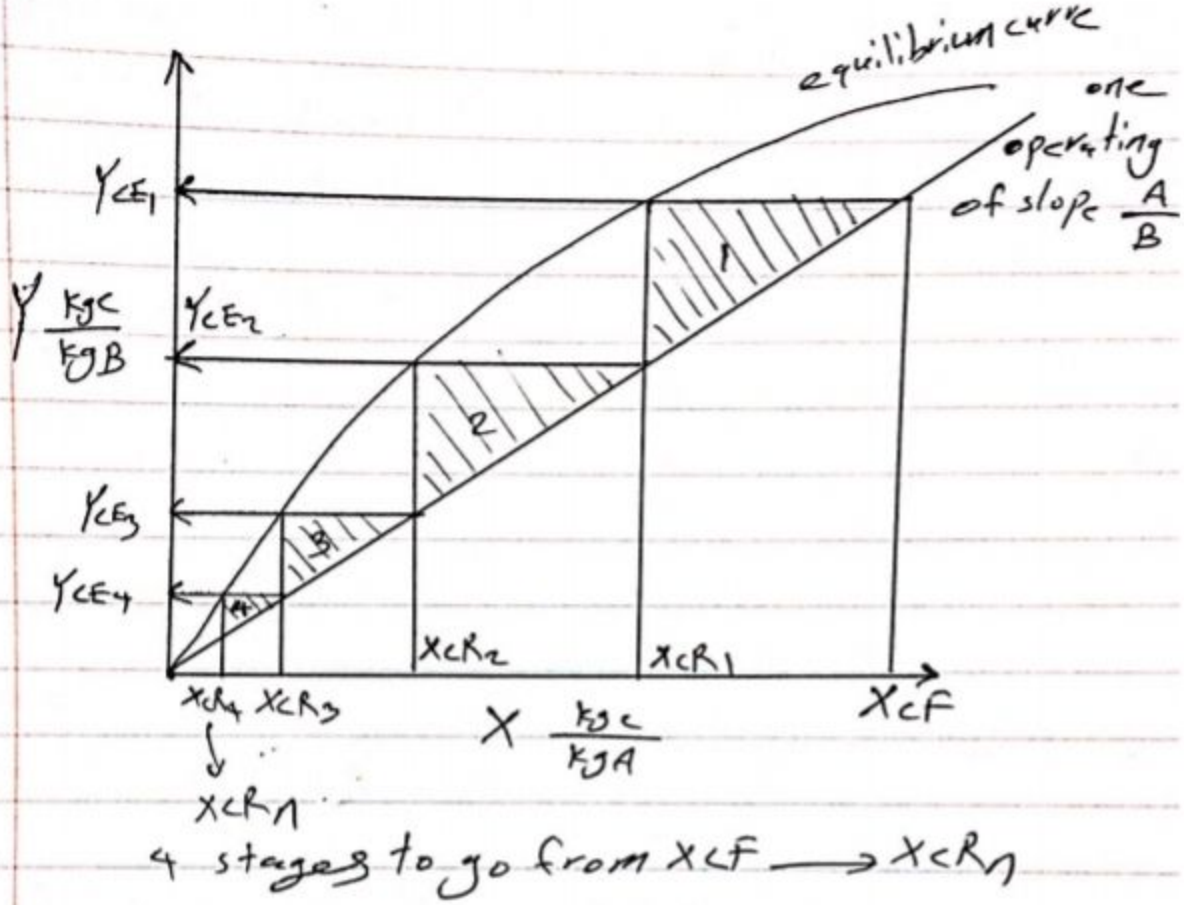
Material Balance on e^{th} stage box-III

$$AX_{CF} + BY_{CE_{e+1}} = AX_{CR_e} + BY_{CE_1}$$

$$\boxed{Y_{CE_{e+1}} = \frac{A}{B}(X_{CR_e} - X_{CF}) + Y_{CE_1}}$$

operating line of slope $\frac{A}{B}$ وهناك خط تشغيل واحد في الاتجاه المعاكس

(13)



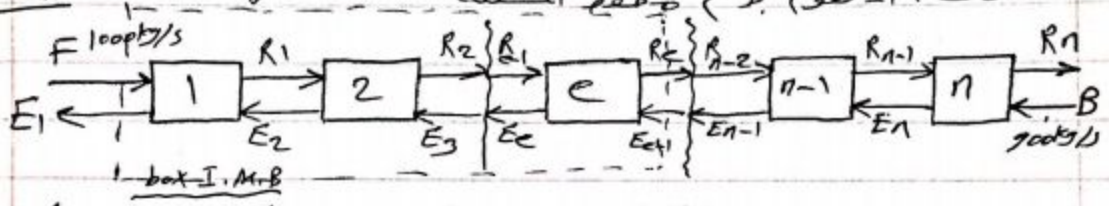
4 stages to go from $X_{CF} \longrightarrow X_{CR_1}$

Example: 1000 kg/s of feed contain 25% C & 75% A with 900 kg/s of solvent B in a counter current extraction system with totaly immiscible between two solvent. How many theoratical stage required to remove 95% of the solute C ?

The equilibrium data are:

X kgC/kgA	0	0.0537	0.233	0.33
Y kgC/kgB	0	0.055	0.29	0.37

Solution: box I



box I, M.B

$$A X_{CF} + B Y_{CE_{e+1}} = A X_{CR_e} + B Y_{CE_1}$$

$$\boxed{Y_{CE_{e+1}} = \frac{A}{B}(X_{CR_e} - X_{CF}) + Y_{CE_1}}$$ → operating line equation

$$X_{CF},\ Y_{CE_1},\ \frac{A}{B},\ X_{CR_n}$$

C in feed = 250 kg/s

A in feed = 750 kg/s

$$X_{CF} = \frac{kgC}{kgA} = \frac{250}{750} = 0.333$$

$$B = 900 \text{ kg/s} \Rightarrow \frac{A}{B} = \frac{750}{900} = 0.833$$

amount of recovered C in extract = $250 \times 0.95 = 237.5 \frac{kg}{s}$

amount of C in final rafinate = $250 \times 0.05 = 12.5$ kg/s

$$Y_{CE_1} = \frac{kgC}{kgB} = \frac{237.5}{900} = 0.263$$

$$X_{CR_n} = \frac{kgC}{kgA} = \frac{12.5}{750} = 0.0167$$

$$Y_{CE_{e+1}} = 0.833(X_{CR_e} - 0.933) + 0.263$$

$$\boxed{Y_{CE_{e+1}} = 0.833 X_{CR_e} - 0.0149}$$

٢- نقوم برسم operating line + equilibrium curve

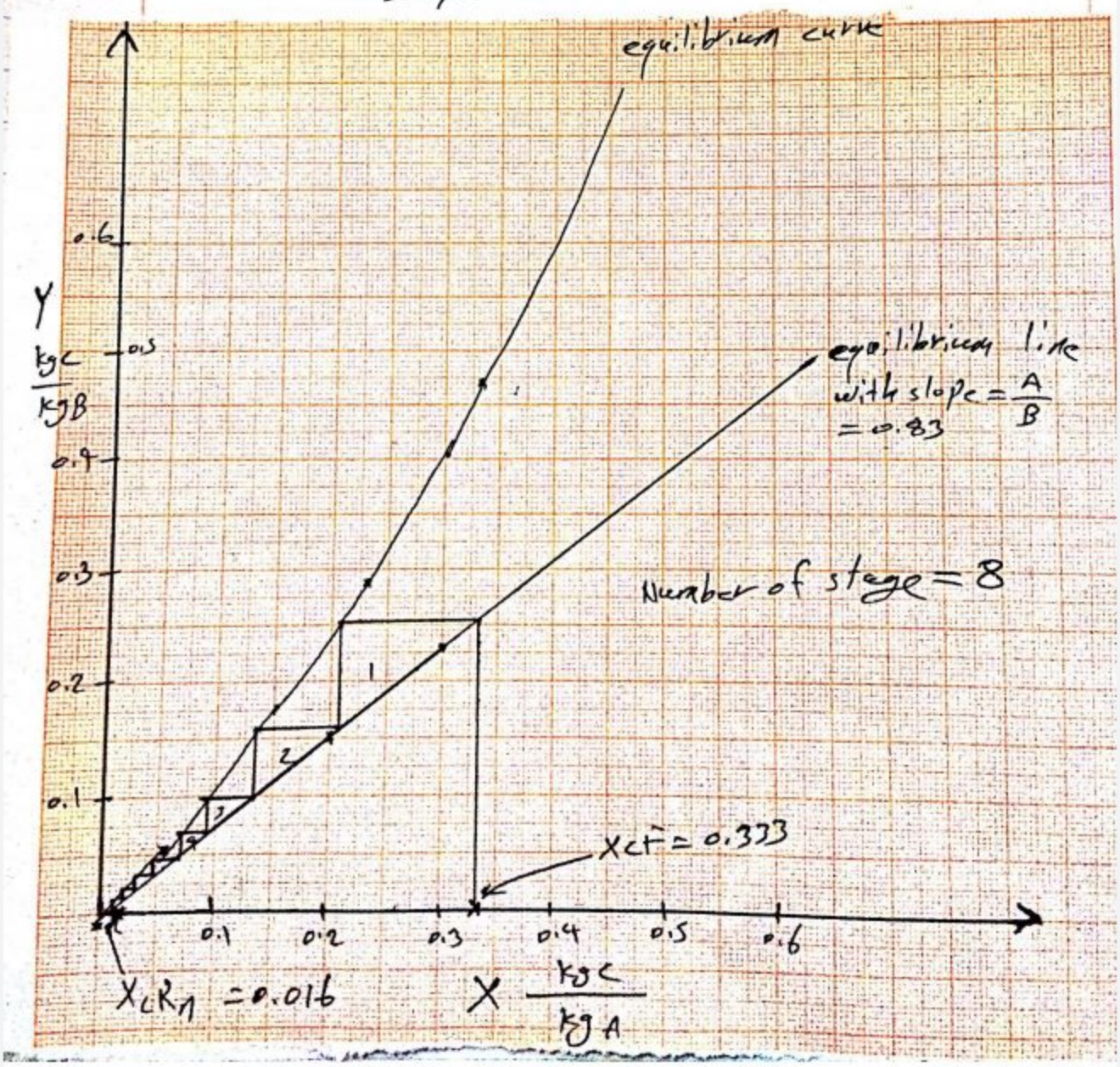
ومنه نحسب عدد المراحل من $X_{CR_1} = 0.0161$ الى $X_{CF} = 0.333$

$$Y_{CE_{n+1}} = 0.833\, X_{CR_n} - 0.0144$$

$X = 0 \Rightarrow Y = -0.0144$

$X = 0.2 \Rightarrow Y = 0.1522$

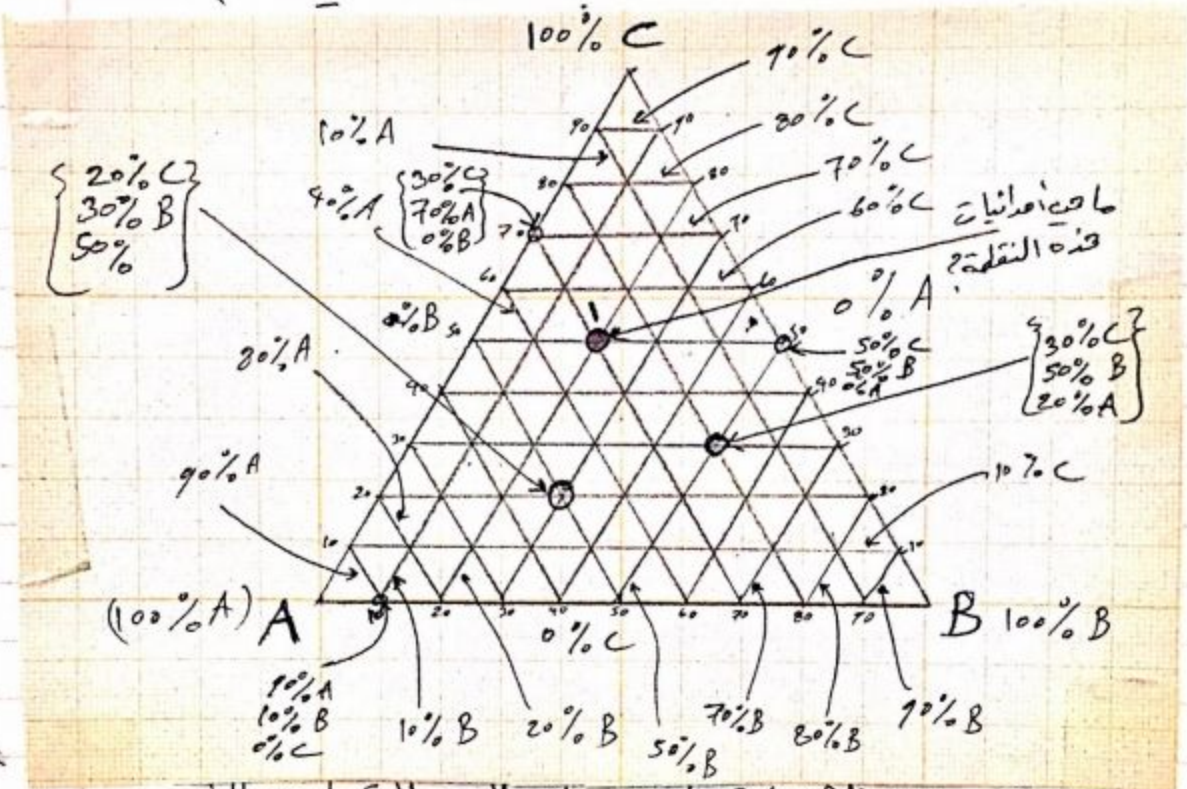
$X = 0.3 \Rightarrow Y = 0.235$



(16)

II) The solvent (B) may be partially miscible with the original solvent (فصل جزئي)

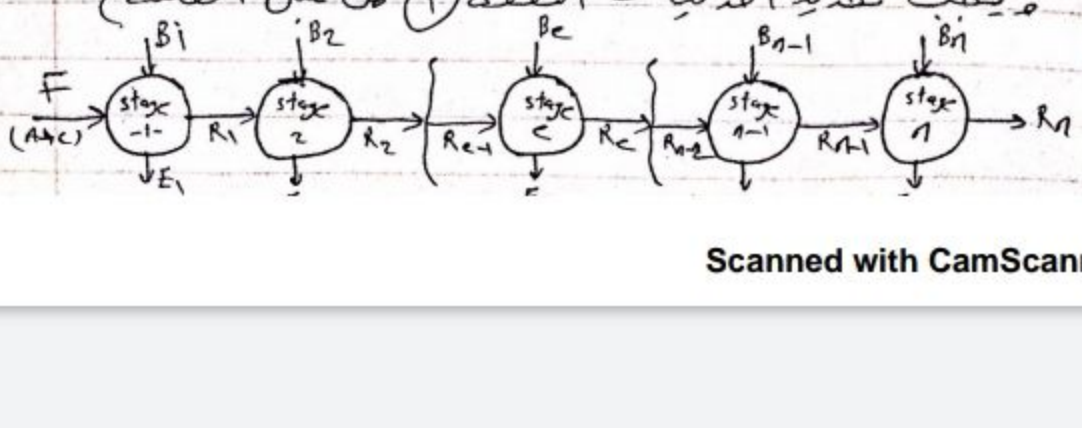
Equilibrium are usually represented by triangular diagram.
أي تكون حالة التوازن متمثلة بالمثلث كما في الرسم أدناه



ماهي أعداد النقطة هذه النقطة؟

يتم تقسيم المثلث المتساوي الأضلاع بالتساوي الى ١٠ أجزاء بحيث أي نقطة داخل المثلث تكون أعدادها متكونة من A ، B ، C أما في أي ضلع من أضلاع المثلث تتكون الأعداد من مادتين فقط لأن المادة الثالثة نسبتها صفر.

يتم متابعة نقاط المثلث بأمعان
ويجب تحديد أعداد النقطة (F) من قبل الطلبة



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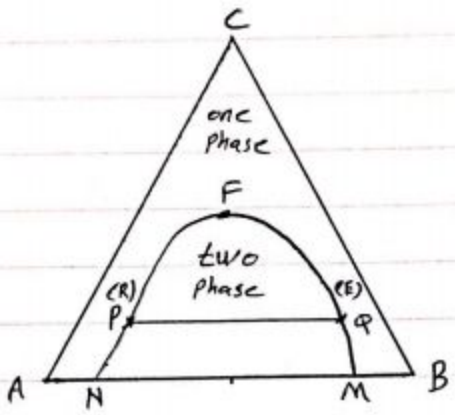


This is the most common type of system in extraction and typical example are water(A) – chloroform(B) acetone (C).

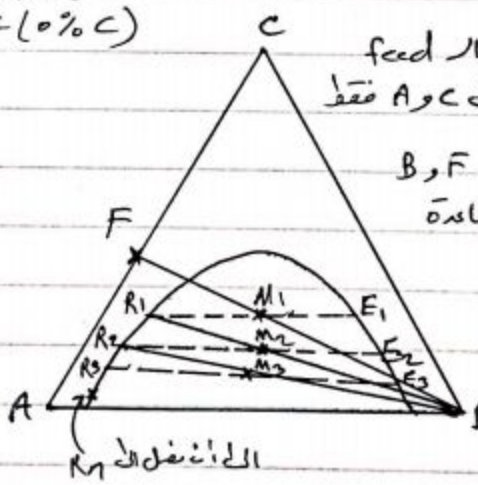
* The solute C is completely miscible with two solvent A & B but two solvent A & B are partially miscible with each other.
* liquid C dissolved completely in A & B but A & B dissolve only to a limited extent in each other to give saturated liquid solution at N (A rich) and M (B rich).
* The distance AN represent the solubility of solvent B in A, and MB represent the solubility of solvent A in B.
* curve NPFQM is the binodal solubility curve. Any mixture outside this curve will be a homogeneous solution of one liquid phase. The area under the curve represents a two-phase region which split up into two layers in equilibrium with each other.

* These layers have composition represented by point P and Q and PQ is known as tie line.
* point F represent a single phase which does not split into two phase which is known as a plait point.
* point F is fixed if either the temperature and pressure is fixed. تبقى نقطة F تقعد الى الاعلى أو تنزل الى الأسفل بتغيير درجة الحرارة او الضغط
* The sid of CB known as Extract (E) → Q
The side of CA known as Raffinate (R) → P

(A) Co-current extraction and solvent A and B are partially miscible فصل جذئي غير تام

The solution is done graphically

1- The solvent B is pure (0% C)



١ - النقطة F تمثل الـ feed الداخل ويحتوي على C و A فقط

٢ - ندسم خط يصل بين F, B

٣ - تحدد النقطة M_1 من قاعدة level rule

٤ - ندسم خط موازي لأقرب Tie line فيتم تحديد R_1 و E_1

٥ - ثم نصل بين R_1B

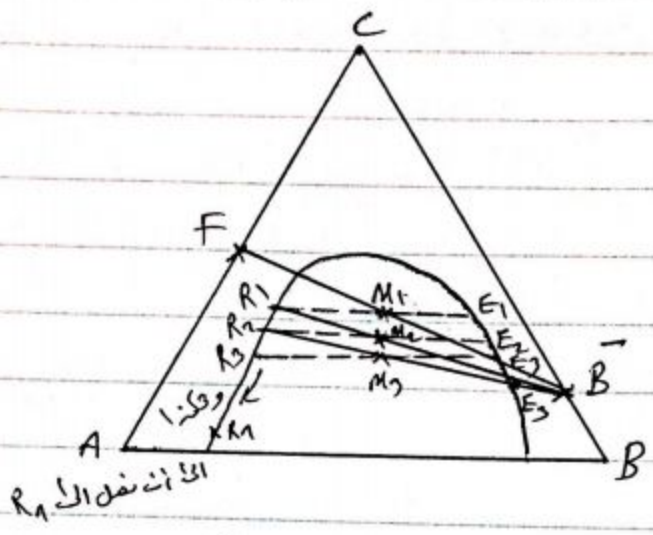
٦ - تحدد M_2 من قاعدة level rule

٧ - ندسم خط موازي لأقرب Tie line فيتم تحديد R_2 E_2

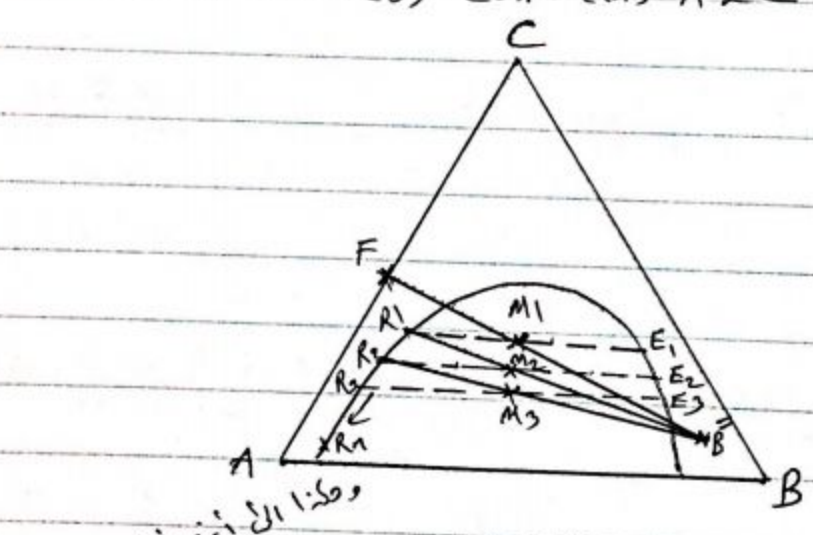
نكرر الخطوات الى ان نصل الى R_M والذي يحتوي على أقل نسبة من C.

(19)

2- The solvent ($\acute{B}$) contains some of C



3- The solvent ($\bar{\bar{B}}$) contains A & C

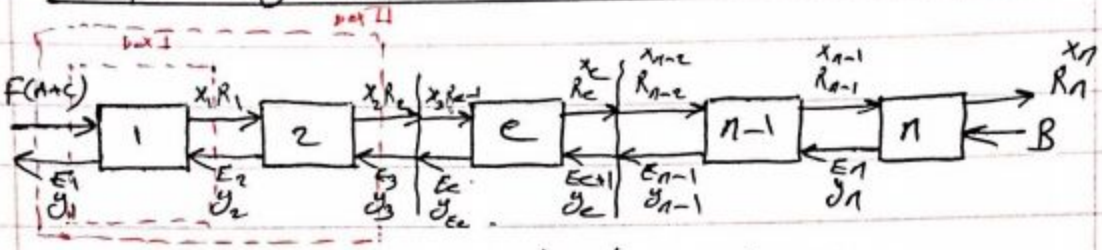


ملاحظة:

بالنسبة لهذا الموضوع فقط السوماتا مقلوبة
ولا توجد أمثلة .

(20)

(B)- Counter current extraction and solvent (B) is partially miscible with solvent A فصل بالمذيبات



Material balance on 1st stage (box I)

$$F + E_2 = R_1 + E_1$$

$$F - E_1 = R_1 - E_2 = P \text{ constant}$$

Material balance on stage (2) (box II)

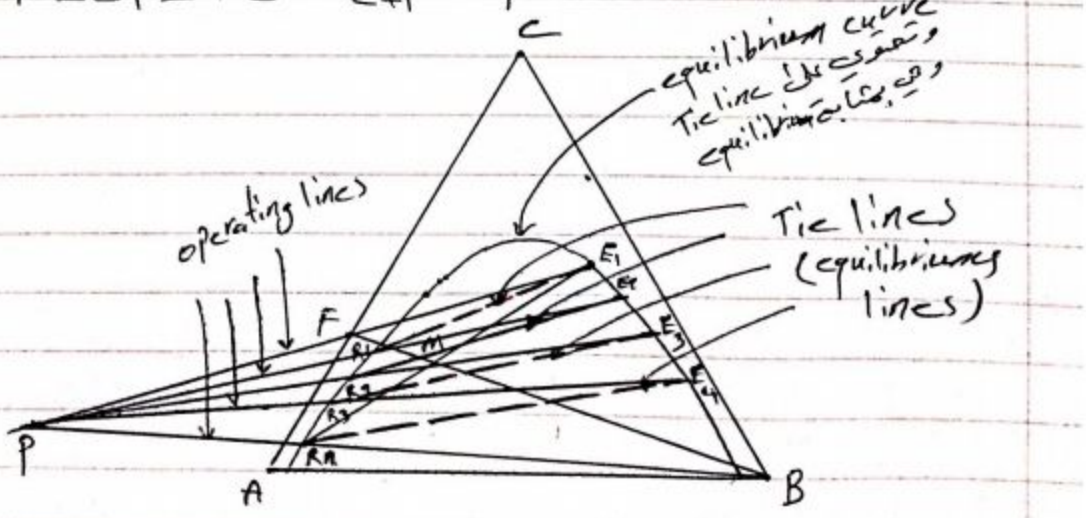
$$F + E_3 = R_2 + E_1$$

$$F - E_1 = R_2 - E_3 = P$$

Material balance on e^{th} stage

$$F + E_{e+1} = R_e + E_1$$

$$F - E_1 = R_e - E_{e+1} = P$$



خطوات الحل :

١- نحدد نقطة F على ضلع AC في المثلث بواسطة معرفة نسبة C , A

٢- نحدد نقطة M وذلك بإيصال المستقيم FB ومن نسبة F و B وقاعدة lever Rule .

٣- تحديد R_1 وذلك من خلال معرفتنا بنسبة الفصل في ال extracter (final %C)

٤- نصل R_1M فيقطع ضلع الـ equilibrium curve في E_1

٥- نحدد E_1 مع أقرب Tieline أو موازي له فنحدد R_1

٦- نجد E_2 بعد PR_1 الى أن يقطع equilibrium curve

٧- نجد R_2 بعد E_2 موازي الى أقرب Tieline فيقطع R_2 عند equilibrium curve

٨- إيصال PR_2 فيقطع Equilibrium عند E_3

٩- إيجاد R_3 بواسطة Tieline أو أقرب Tieline موازي وهكذا الى أن نصل الى R_1 وعندها نحسب عدد المراحل

ملاحظة مهمة :

لتحديد نقطة P وذلك بعد FE_1 و R_1B فيتقاطعان عند نقطة P وأحياناً تكون يميناً أو يساراً .

Example
(1)

Example 13.2

A 50 per cent solution of solute **C** in solvent **A** is extracted with a second solvent **B** in a countercurrent multiple contact extraction unit. The mass of **B** is 25 per cent of that of the feed solution, and the equilibrium data are as given in Figure 13.17.

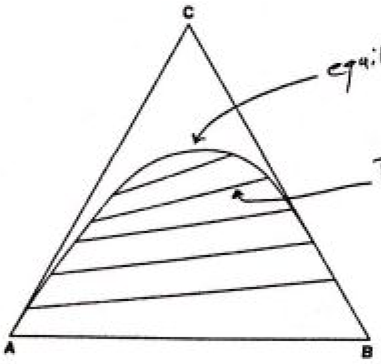


Figure 13.17. Equlibrium data for Example 13.2.

Determine the number of ideal stages required and the mass and concentration of the first extract if the final raffinate contains 15 per cent of solute **C**.

Solution

The equilibrium data are replotted in Figure 13.18 and F, representing the feed, is drawn in on AC at $C = 0.50$, $A = 0.50$. FB is joined and M located such that FM/MB = 0.25. R_n is located on the equilibrium curve such that $C = 0.15$. In fact $B = 0.01$ and $A = 0.84$. E_1 is located by projecting R_nM on to the curve and the pole P by projecting E_1F and BR_n. R_1 is found by projecting from

خطوات الرسم وحساب عدد المراحل

١- تحديد F على خط CA وذلك من خلال نسب (50%A) و (50%B) أي في المنتصف.

٢- تحديد نقطة M بواسطة قاعدة level Arle
$(25\% B)(75\% F) \Rightarrow \frac{FM}{MB} = 0.25$

٣- نحدد R_n (C 15%) ونحدد موقعها على equilibrium curve.

٤- نصل R_nM فيتقطع equilibrium curve عند E_1

٥- نحدد نقطة P وذلك بإيصال E_1F و BR_n فيتقاطعان عند نقطة P.

٦- إيجاد R_1 بعد E_1 خلال Tie line أو اقرب Tie line موازي له فيقطع equilibrium curve عند R_1.

٧- نحدد E_2 بعد PR_1 الذي يقطع equilibrium curve عند E_2.

٨- نحدد R_2 بعد E_2 بأقرب Tie line فيقطع equilibrium curve عند R_2.

٩- بإيصال PR_2 فيتقطع Equilibrium curve عند E_3

١٠- إيجاد R_3 بواسطة أو اقرب موازي له Tie line وهكذا إلى أن نصل إلى R_n

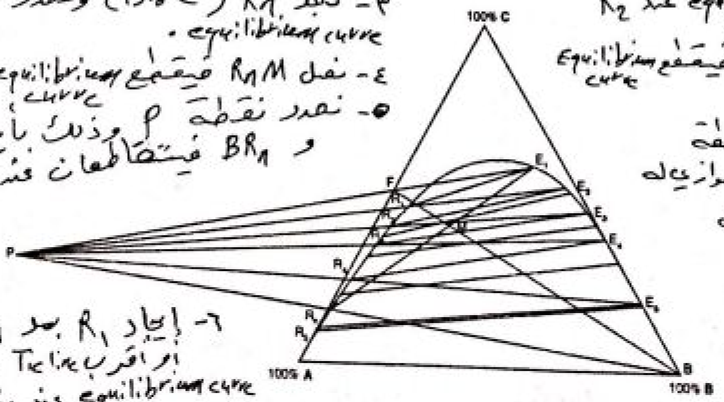


Figure 13.18. Graphical construction for Example 13.2.

E_1 along a tie-line and E_2 as the projection of PR_1. The working is continued in this way and it is found that R_5 is below R_n and hence 5 ideal stages are required.

From Figure 13.18 the concentration of extract E_1 is 9 per cent **A**, 58 per cent **C**, and 33 per cent **B.**

example (2)

(23)

It is proposed to reduce the concentration of acetaldehyde in aqueous solution from 50 per cent to 5 per cent by mass, by extraction with solvent B at 293 K. If a countercurrent multiple-contact process is adopted and 0.025 kg/s of the solution is treated with an equal quantity of solvent, determine the number of theoretical stages required and the mass flowrate and concentration of the extract from the first stage.

The equilibrium relationship for this system at 293 K is:

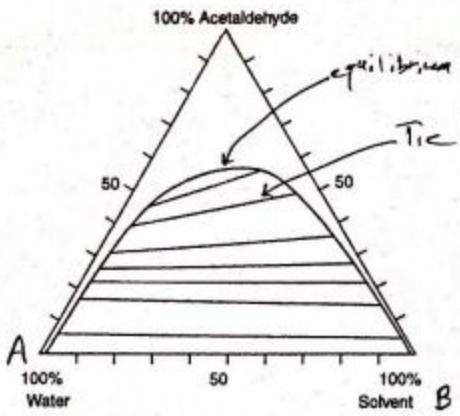


Figure 13a. Equilibrium data for Problem 13.9

Solution

The data are replotted in Figure 13b and the point F, representing the feed, is drawn in at 50 per cent acetaldehyde, 50 per cent water. Similarly, R_n, the raffinate from stage n located on the curve corresponding to 5 per cent acetaldehyde. (This solution then contains 2 per cent S and 93 per cent water.) Fβ is joined and point M located such that FM = M , since the ratio of feed solution to solvent is unity. R_nM is projected to meet the equilibrium curve at E_1 and FE_1 and $R_n\beta$ are projected to meet at P. The tie-line E_1R_1 is drawn in and the line R_1P then meets the curve at E_2. The working is continued in this way and it is found that R_4 is below R_n and hence four theoretical stages are required.

في هذا المثال نجد نقطة لم تقع على يمين الرسم بينما في السؤال السابق تقع على يسار الرسم

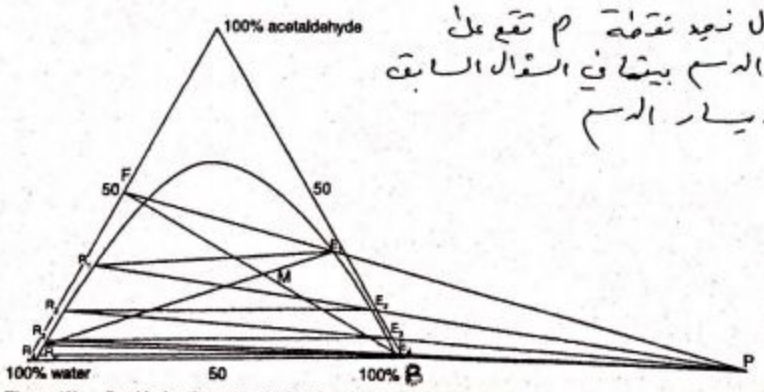


Figure 13b. Graphical construction for Problem 13.9

From Figure 13b, the composition of the extract from stage 1, E_1, is:

3 per cent water, 32 per cent acetaldehyde, and 65 per cent

Making an overall balance, $F + \quad = R_n + E_1$

Thus: $(0.025 + 0.025) = (R_n + E_1) = 0.050$ kg/s

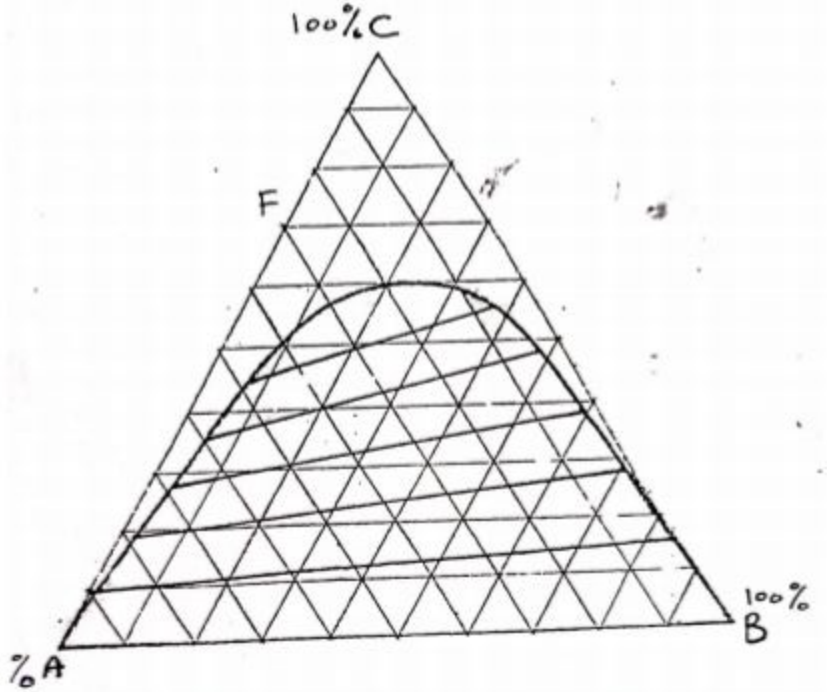
Making an acetaldehyde balance:

$$(0.50F + 0) = (0.05R_n + 0.32E_1)$$

$$0.05(0.050 - E_1) + 0.32E_1 = (0.50 \times 0.025) \quad \text{and} \quad E_1 = 0.037 \text{ kg/s}$$

(24)

أرجو من الطلبة حل المثال التالي على هذه الورقة



(25)

أرجو منك طلبة حل المثال الأول على هذه الورقة