Mass Transfer Operations II

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Lecture 15

Solid-liquid Extraction

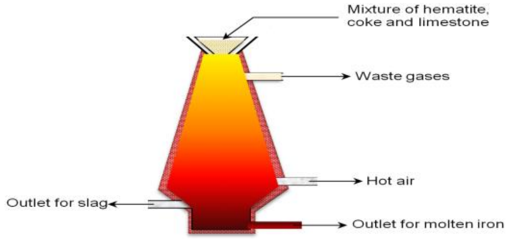
Welcome back to mass transfer operation 2 course and now we will be discussing on the new topic solid-liquid extraction or we can say this is leaching process.

1. Leaching

Solid liquid extraction or leaching is the process of separation of soluble constituents of a solid material using a suitable solvent.

Example: *(i)* Extraction of oil from oil seeds – Oil is product and solid residue is by product.

(ii) Metallurgical Industries – Metal extraction from ores.



Basic steps of leaching/solid-liquid extraction:

1. Intimate contact between solid feed with solvent.
2. Separation of extract from exhausted solid.
3. Separation of solvent (and entrained solid) from extract followed by purification of the product.
4. Recovery of solvent from moist solid (by pressing / squeezing and drying).

- In leaching, a substantial part of solid does not dissolve and soluble matters diffuse out through the solid.
- An acid, alkali or solution of a complexing chemical is commonly used for solubilizing the target materials.

- Leaching of ores (oxides, carbonates, sulphides etc.,) is a major step in hydro metallurgy.
  - → Leaching of copper minerals by H_2SO_4 or ammonical solution.
  - → Leaching of Au from to ore by NaCN solution.
  - → Leaching of low grade Ni ore, nickel laterite with H_2SO_4 at 250°C and 650psi. Co is by product.
- Bio leaching by bacteria like Thiobacillus Ferro oxidans and thermophilic species such as sulphobacillum, Acidianus are well known. These convert ores into soluble sulfonates. Bioleaching is slow but non-polluting since no SO_2 is emitted. Cu, Au, Zi and Ni are separated by bioleaching.

Solid Liquid extraction equilibrium

Concentration of solute in clear liquid = Overflow

Fraction of liquid in slurry = Underflow

Notation:

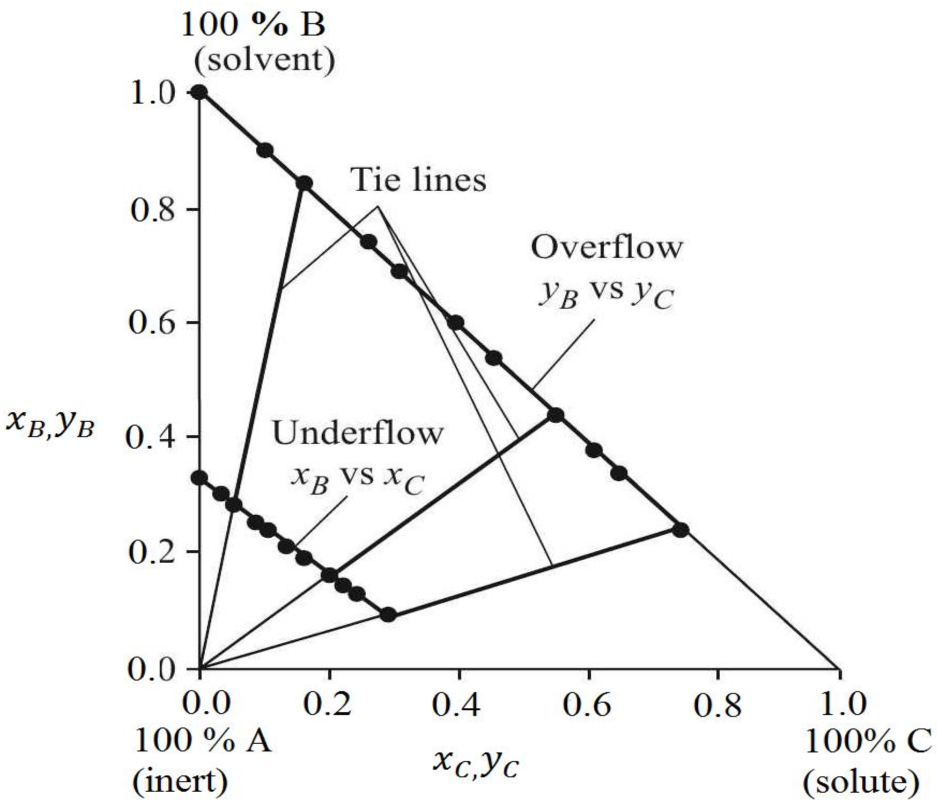
Mass fraction of species in the overflow = y

Mass fraction of species in the slurry = x

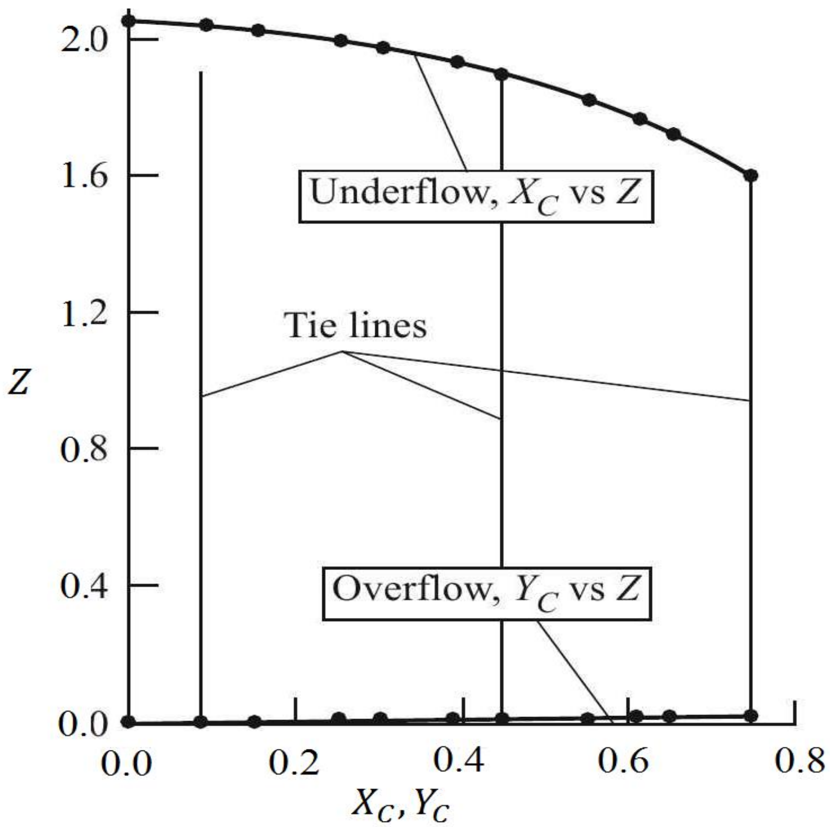
- ➢ Two common techniques for representing solid – liquid extraction equilibrium data:

1. The triangular diagram
2. The Ponchon – Savarit Diagram

1. The triangular diagram:

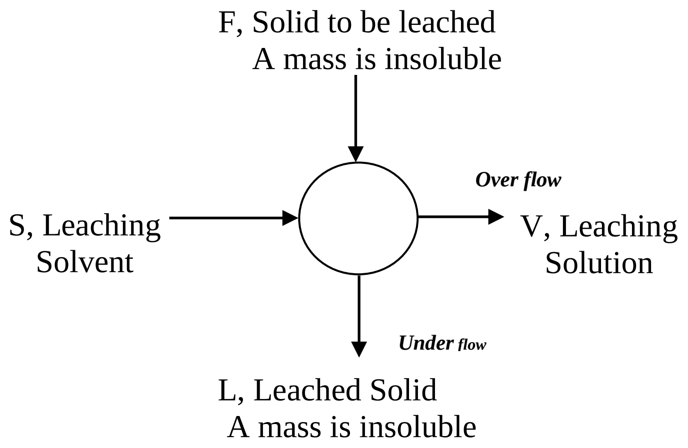


2. The Ponchon – Savarit Diagram:



2. Leaching Operations

a) Single – Stage Leaching:



And this is first operation that is single stage leaching operation so suppose this is the leaching process so we have added this F with A amount of mass that is insoluble or we can say this is nothing but inert. So insoluble solid carrier that is A, it is equivalent to that of the carrier solvent like there in case of this extraction A. And then pure solvent whatever is added that is B, so here pure solvent is added this B but we are adding this S amount of this pure solvent we can say this one leaching solvent because it does the leaching process, leaching is nothing but coming out of the solute whose through from the solid material.

b) We are allowing this process to reach tis equilibrium then it will be converted into two phases, one will be the overflow that is our target, where overflow is V and one is this underflow that is not require or it will give this by-product. So that is we can say this one underflow or we can say this leached solid or exhausted solid.

c) But we need to remember that the whatever the target component C is there say soluble solute or target component will be divided into two parts like this one major part will go to overflow and minor part will come to or will be carried through this inert insoluble solid or which is inert. So we cannot completely remove this target component this one from this inert because there will be some amount of association of this target component with the carrier solid A.

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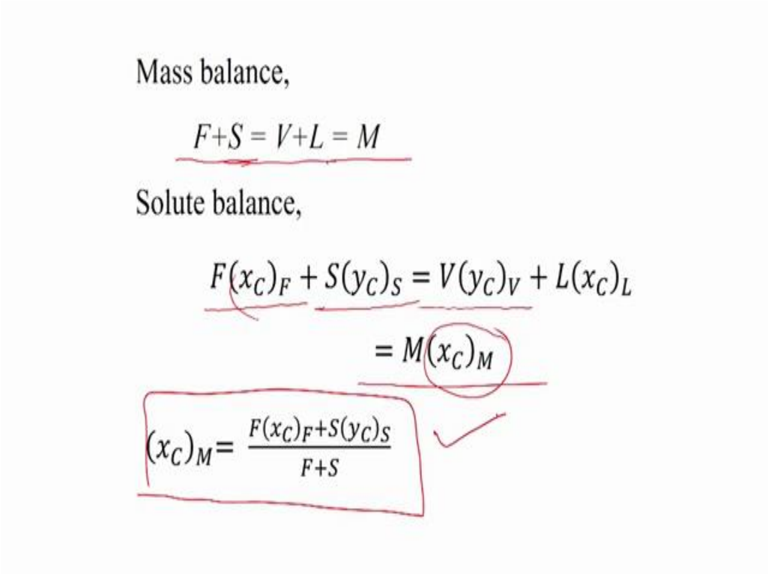

e)

Let, Insoluble carrier solid = A

Pure solvent = B

Soluble solute = C

$$N = \frac{\text{insoluble weight}}{\text{soluble weight}} = \frac{A}{B+C}$$

Mass balance, F+S = V+L = M

Solute balance, $F(x_c)_F + S(y_c)_S = V(y_c)_V + L(x_c)_L = M(x_c)_M$

$$(x_c)_M = \frac{F(x_C)_F + S(y_C)_S}{F+S}$$

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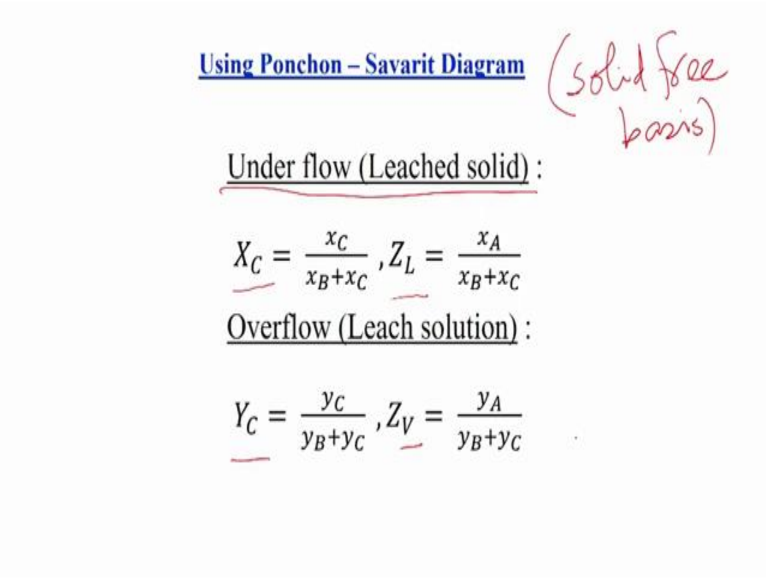

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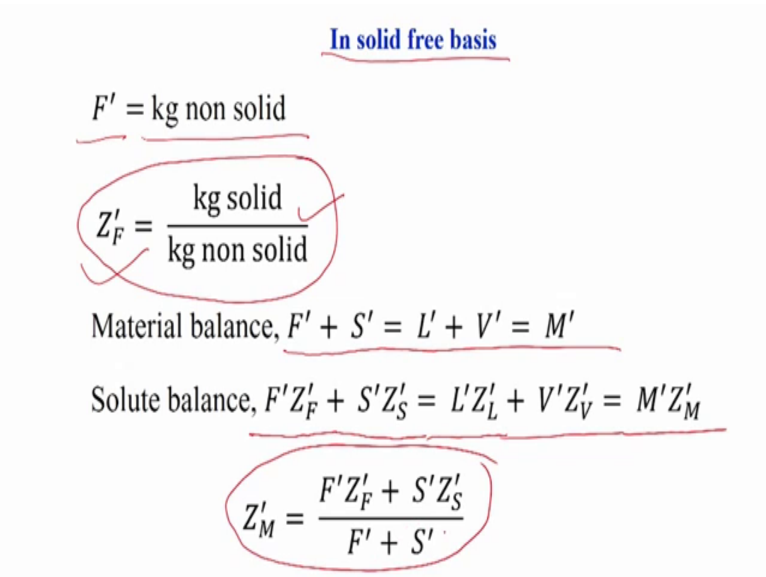

g)

Under flow (Leached solid) : $X_C = \frac{x_C}{x_B+x_C}, Z_L = \frac{x_A}{x_B+x_C}$

Overflow (Leach solution) : $Y_C = \frac{y_C}{y_B+y_C}, Z_V = \frac{y_A}{y_B+y_C}$

F' = kg non solid

$$Z'_F = \frac{\text{kg solid}}{\text{kg non solid}}$$

Material balance, $F' + S' = L' + V' = M'$

Solute balance, $F'Z'_F + S'Z'_S = L'Z'_L + V'Z'_V = M'Z'_M$

$$Z'_M = \frac{F'Z'_F + S'Z'_S}{F' + S'}$$

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Problem 1:

One thousand kg of crushed oil seeds (19.5% oil + 80.5% meal) is extracted with 1500 kg pure hexane in a a batch extraction vessel. Calculate fraction of oil extracted using

a) Right triangular diagram.

b) Ponchon – Savarit diagram.

Equilibrium data as follows:

Over flow (100 kg), solution			Underflow (100kg), slurry		
W_A (kg), y_A	W_B (kg), y_B	W_C (kg), y_C	W'_A (kg), x_A	W'_B (kg), x_B	W'_C (kg), x_C
0.3	99.7	0.0	67.2	32.8	0.0
0.45	90.6	8.95	67.1	29.94	2.96
0.54	84.54	14.92	66.93	28.11	4.96
0.70	74.47	24.83	66.58	25.06	8.36
0.77	69.46	29.77	66.26	23.62	10.12
0.91	60.44	38.65	65.75	20.9	13.35
0.99	54.45	44.56	65.33	19.07	15.6
1.19	44.46	54.35	64.39	16.02	19.59
1.28	38.50	60.22	63.77	14.13	22.10
1.28	34.55	64.17	63.23	12.87	23.90
1.48	24.63	73.89	61.54	9.61	28.85

Solution:

(a) F = 1000kg, S = 1500kg

$(x_C)_F = 0.195$, $(y_C)_S = 0$

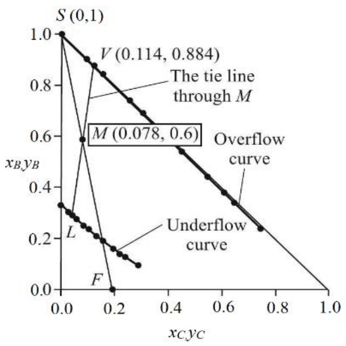
M = F+S = 2500kg

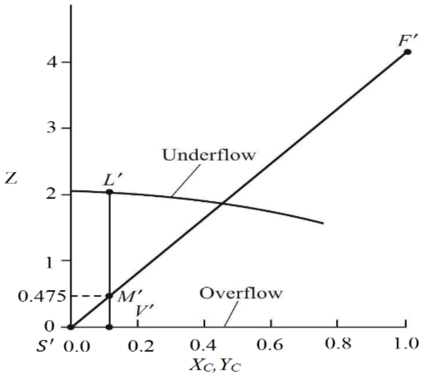
$$(x_C)_M = \frac{1000 \times 0.195 + 1500 \times 0}{2500} = 0.078$$

F ($x_A = 0.805$, $x_B = 0$, $x_C = 0.195$)

S ($y_A = 0$, $y_B = 1$, $y_C = 0$)

Join FS. Point M represents a mixture of F and S at $(x_C)_M$= 0.078





$$V(y_C)_V + L(x_C)_L = M(x_C)_M$$

$V \times 0.114 + L \times 0.0403 = 2500 \times 0.078 = 195$

V+L = 2500

V = 1279kg; L = 1221kg

Amount of oil extracted = $V(y_C)_V = 1279 \times 0.114\ kg = 145.8kg$

Initial amount of oil in feed = 1000 × 0.195 = 195kg

∴ Fraction extracted = $\frac{145.8}{195} \times 100\% = 74.8\%$

(b) $F' = 1000 \times 0.195kg = 195\ kg = kg\ of\ non\ solid$

$X_{C}{'}_F$ = mass fraction of solute in feed on solid free basis = $\frac{195}{195} = 1$

$$Z'_F = \frac{kg\ solid}{kg\ non\ solid} = \frac{805}{195} = 4.13$$

$S' = 1500kg, (Y_C)_{S'} = 0\ (pure\ solvent), Z'_S = 0$

M' = 195+1500 = 1695;

$$Z'_M = \frac{F'Z'_F + S'Z'_S}{F' + S'} = \frac{195 \times 4.13 + 1500 \times 0}{1695} = 0.475$$

F'(1, 4.13), S'(0, 0), M' is located on line $S'F'$ at Z'_M = 0.475

Z'_L = 2.03, Z'_V = 0, $(X_C)_{M'} = (X_C)_{L'} = (Y_C)_{V'} = 0.115$[same vertical line]

$L'Z'_L + V'Z'_V = M'Z'_M$;

$L' \times 2.03 + V' \times 0 = 1695 \times 0.475$

L' = 396.6kg, V'= (1695-396.6) kg = 1298.4kg

Mass of overflow = 1298.4kg

$\therefore$ Fraction of oil extracted = $\frac{V'(Y_C)_{V'}}{F'} \times 100 = \frac{1298.4 \times 0.115}{195}\% = 76.6\%$

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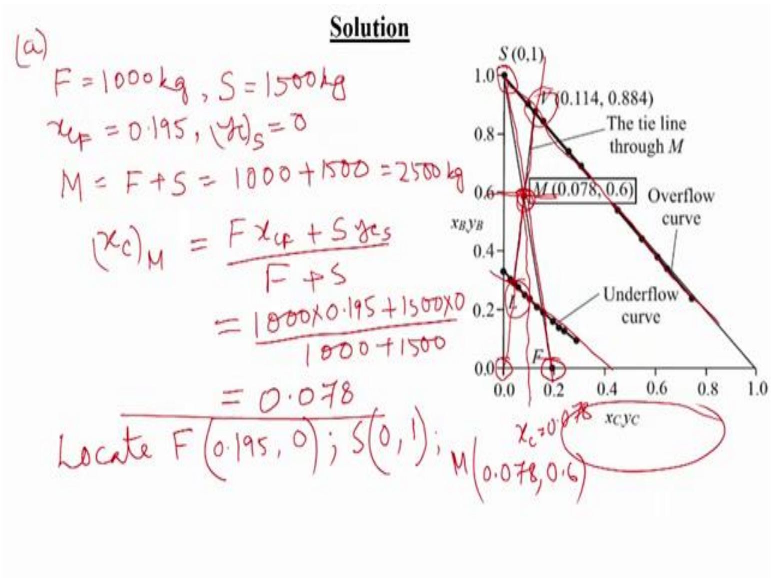

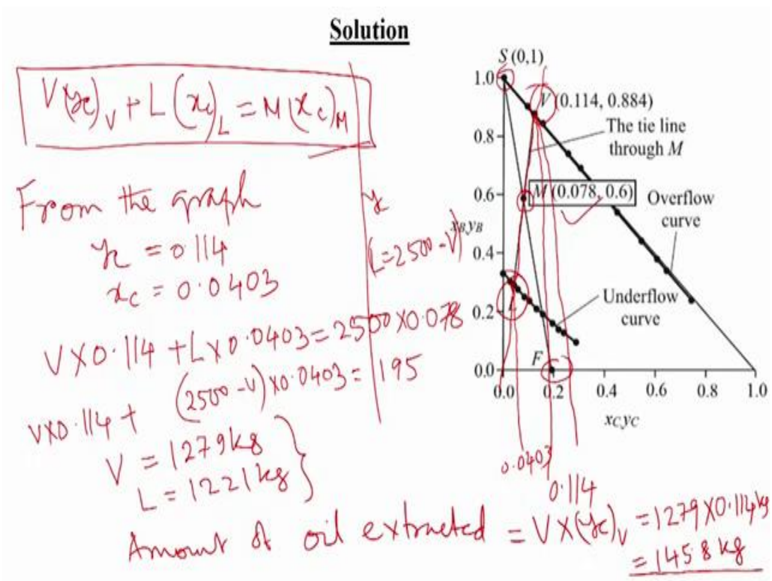

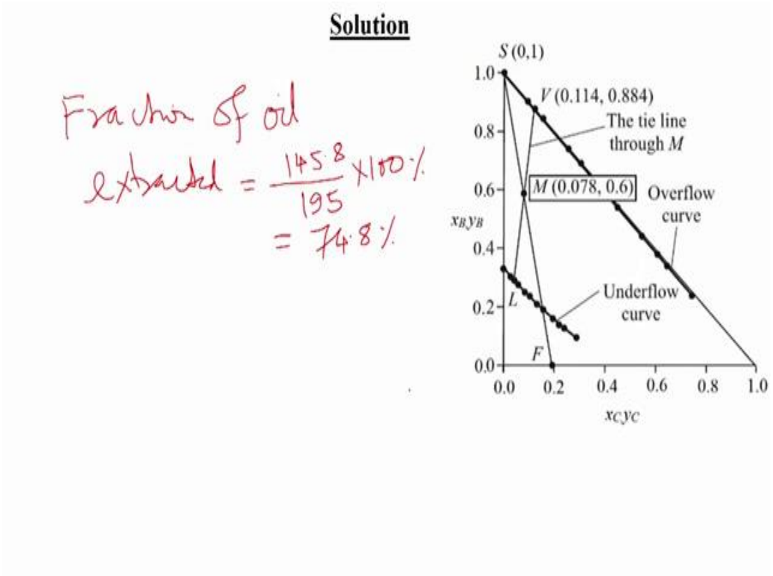

So we will start solving this one, whatever the data points are given already will be first using those and then we will be placing all these in the right triangular diagram first we will try this right triangular diagram then we will be doing this for the Ponchon – Savarit or the solid free basis we will be doing that. So for this whatever the data points are there already we will need to get this one say first feed is given as 1000 kg and this solvent is given as 1500 kg. And then xCF that is given as 19.5 percent so that will be 0.195 and whereas this yCS that is in that case it is 0 because it a pure hexane is given, so it is, it has no in the contribution toward this oil extraction.

So we have this, now mixture is equal to F plus S that is will be like 2500 kg. Now we can get this say xCM that will be like F xCF plus S say yCS by F plus S, so it is coming out as 1000 into 0.195 plus 15000 into 0 divided by 1000 plus 1500. So it is coming out as very small value 0.078. So we will now be getting this three points like this, so first we need to locate, so first step will be locate, this F that is F will be like this in the C axis. So every time actually we put this one F in the C axis because it contains only A and C, so there you know B because you see A is the inert or carrier solid and C is the target component.

So it is 19.5, so we can day F will be like this 0.195 almost here with this yB that is equal to 0. So this is coming out here. And then we have this S, for S xC is 0 or yC whatever is that is 0 and that it is a pure one, so yB is equal to 1. So this is nothing but this point. First we need to join this S and F, I am not repeating the procedure or design calculations for this leaching unit because this is the same as we followed in case of this liquid-liquid extraction. So the same procedure actually we need to follow. So we need to join this S and F and now we have this xCM that is 0.078, so somewhere it will be like this one, so this one 0.078, so will be coming to this point say xC is equal to 0.078, so then we will be getting this M.

So now we have this S and M is like this say mixture point actually will be like this 0.078 and from here actually we will be getting as this around say Y will be like 0.6, so it is like here, so we are getting this M here so that is place in this line. Now the procedure is very simple like this one, whenever we have this M, I mentioned that if we just draw a straight line from this 0. 0 point, so then it will be like this say if we start from here and if we extent this one through M will be get, it will touch this we can say this overflow line and this underflow line, it will touch, this means if we keep this mixture for a long period of time then it will be converting into this underflow and overflow.

This overflow line means it will be touching at V, so we can say this one we have this, now it is converting, now this F we have, we have S and we have mixture. Now we are drawing the straight line, that is nothing but the I told that this is the tie line for this one and now it is converting into V and L. So here this from this diagram actually we will be getting all this composition like this, so here if we do the component balance here say like this V into yCV plus L into say xCL, that will nothing but say M into xCM.

So here this mixture is here actually we say 2500, so now we can do one thing, so from here, so whenever we will be using this graph paper we will find that, for this L we will be getting like this from this graph or we can say right here from the graph say yC will be like this, this value actually is 0.114 and xC that is obtained as xC you see this sorry, this V into yC this one, that is obtained as that is 0.114 and for xC it is obtained as 0.0403, so this is 0.0403.

So we will be this one getting this one from this graph paper will be easily getting this one and now when we putting this one in the component balance, from where we will be getting both L and V. So we will be putting that V into yC that is nothing but 0.114 that is in the overflow line plus L into 0.0403 that is equivalent to 2500 that is the mixture into xCM is obtained as 0.078 and we know this L is equal to 2500 minus V, that it L is equal to 2500 minus V.

So we will be putting now say V into 0.114 plus 2500 into 0.0403 is equal to say 2500 into 0.078, so that is coming out as 195. So from here we will be getting V is equal to 1279 kg and say L is equal to we can say 2500 minus 1279, so it is coming out as 1221 kg. So now, so the amount of, amount of oil extracted that is V into yCV that is coming out as V is equal to 1279 into yC is already obtained from this graph, that is 0.114 kg, so it is coming out as 145.8 kg.

So now we have this, that amount this 145.8 kg oil in that overflow, so from here the fractional extraction or fraction of oil extracted is equal to total oil was actually say out of 1000 kg say 19.5 percent, that is nothing but 195 kg, so out of this 195 kg, that 145.8 kg is recovered or extracted, so it is percentage so it coming out as 74.8 percent. So using this single stage operation the 74.8 percent is extracted from the extraction, this leaching unit. So there 15000 kg hexane is used for the separation of oil from 1000 kg feed or solid.

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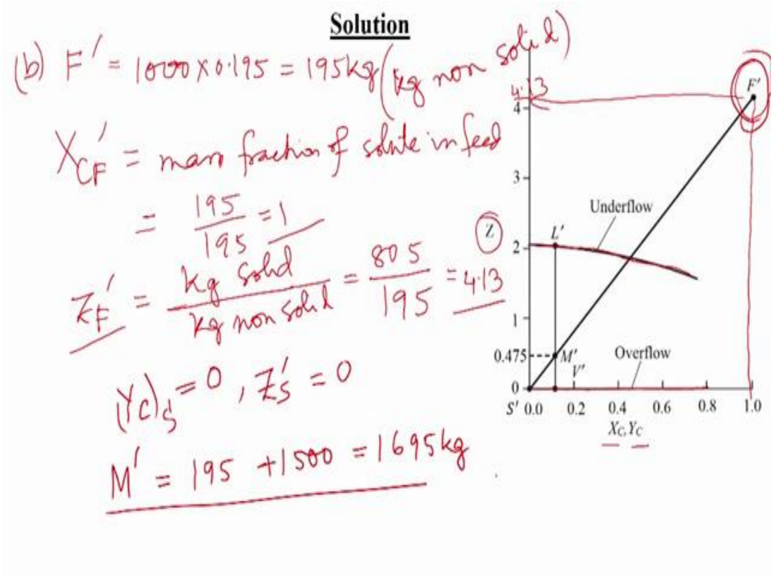

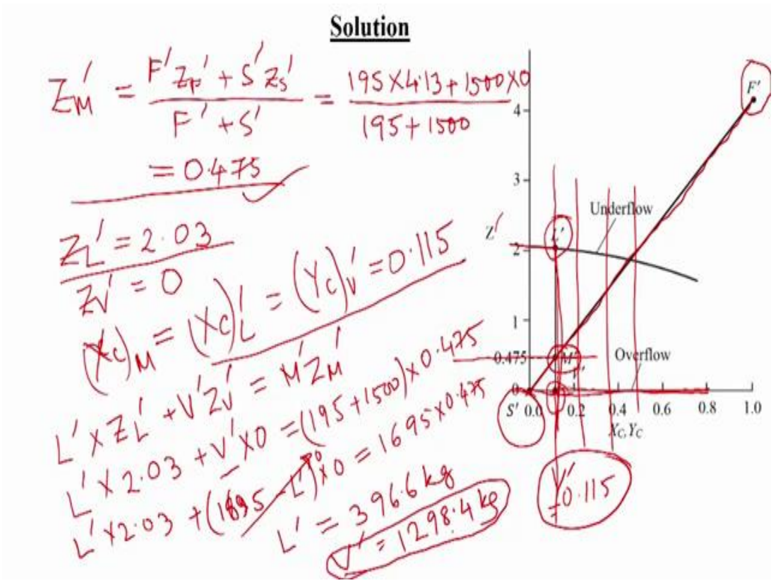

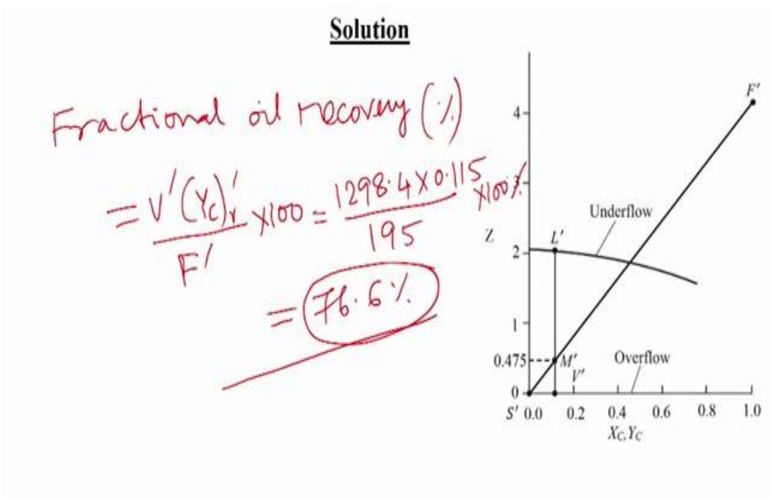

Now we will be discussing with the using this Ponchon – Savarit diagram. So I told that say whenever we will be this one solving this problem using this Ponchon – Savarit, this is the second way of (()) (36:20) solving the problem. So there will be converting this overflow and underflow in terms of capital XC capital YC and Z, so Z values, so it will be getting this one, so now ZL and ZV, so we will be putting this one and then we will be getting the underflow and overflow line. And I told that, this in case of the underflow the in most of the cases the values will be more than 1.

So that is, mostly it is the inert materials or where the cakes will be there or we exhaust solid will be there and this F prime whatever the feed actually has this Z value as a more than this one what is called 4 here, 3 in the particular case. So now here we will be doing this one in terms of solid free basis like this F prime will be like this, so out of this whatever the if we if we remove this inert solids, only 19.5 percent solubilized solid is there, then it will be like this 1000 into 0.195 that is it will be 195 kg.

So this is F prime in 195 kg, that is it is simply nothing but kg non-solid, so this is called kg non-solid and then if we say xC F prime actually that will be like this mass fraction, mass fraction of solute. So in feed, so that will be, whenever it is solute in feed and that is also in non- solid basis, so it will like this 195 by 195, so that is nothing but say 1. Because you see this in total solubilized solid, how much amount of this one target component is there, say that is 195 by 195.

So whenever we will be discussing xCF that is in all the cases it will be like 1. So xCF that will be arise like 1, xCF. And then say ZF prime actually, so that we derived as kg solid by kg non-solid, so that is the convention. So this here actually the kg solid is means this out of this 1000 kg 195 kg is a kg non-solid, so it is 1000 minus 195 that is coming out at 805.

So it is coming out as 4.13, that is why you see ZF prime or say the value Z value of this feed is more than 4, so in that case. So as we have this one, this is 4.13, so that is we can say this one and for this whenever we can say, we can say this one when xCF is equal to 1 or say capital XC or YC whatever that is for this case, that is 1. So this is feed point location is placed here, so feed point location is placed here now, now we can say this yC S prime that is here you see this one, the same problem here solvent is pure hexane.

So it is equal to say yC S prime that is 0 and again say ZS prime also that will be 0, so because here this non-solid means that is target component that is not present in the solvent, so it is 0 also. Now this M prime will be like this say total mixture that will in the solid free basis, so from this feed we will getting say 195 kg and this from the extracting solvent, we will be getting the entire one 1500 because this is the pure non-solid or we can solvent. So it is coming at 1695 kg.

So total mixture is like this, so now we will be say calculating this ZM prime, so ZM prime is nothing but it is from the formula A prime and ZF prime plus S prime into ZS prime by this F prime plus S prime. So it will be like this, F prime is 195 into ZF prime is whatever we got this one 4.13 plus S prime that is 1500 into ZS prime is equal to 0 divided by 195 plus 1500.

So it is coming out as 0.475, so you see this one in this case, whatever the mixture is there so we have now located this feed point, we have located this S prime that is nothing but 0. 0 because it does not contain any target component. So now again we need to join this F prime and S prime as usual then we need to get whatever the ZM prime is this one 0.475, so for that case we will be getting M prime here and say this one say M prime will be obtained when Z prime will be 0.475 like this, these are a Z prime.

Now you see this F is located then S prime is located and M prime is located, now you see this one all the tie line values are actually now vertical, whatever the tie lines values actually we will be getting they are all will be vertical. Because here we have assumed, we are expressing this we can say underflow and overflow with the solid free basis, that is why all the tie lines are vertical.

So now we need to this one draw this vertical line through M prime that is lying in between S prime and F prime. So then we will be getting this L prime and we will be getting here this V prime. So we will be, this one, now we will be getting this V prime and L prime, so they are actually getting this ZL prime that is actually is, just above this two, that is obtained from this graph actually it is obtained as 2.03.

And say ZV prime there is obvious that it will be almost 0 because you see this overflow line, whenever we will be converting this into solid free basis, in the overflow because there will be solid, there will be solid, so that is why it is, always it will be lying in the x-axis. So that is why ZC prime will be 0. Now we will be doing this one say, we can say this XCM whatever the values actually we are getting this xC value actually for this, that is for all the cases, we can say this one for underflow, for overflow, for mixture, that is XCM value that is capital XCM value is nothing but the XCL prime that is nothing but YCV prime.

That is coming out as this one this is 0.115, so it will be, if you use this graph paper then we will be getting this Y prime as 0.115, so this is we can say 0.115. So now we will be doing, again we will be doing this component balance like this we will be getting L prime into ZL prime plus V prime into ZV prime that will be coming as M prime into ZM prime.

So here, L prime into ZL prime that is coming out as ZL prime, that is we can say ZL prime 2.03 so that is 2.03 plus V prime into ZV prime that is we got as 0 is equal to M prime that is M prime is nothing but 195 plus 1500 into ZM prime that we got as 0.475, .0457 and here also V prime is equal to this, this 195 plus 1500 that is 1695 minus L prime.

So we can say this one L prime into 2.03 plus 1695 minus L prime into 0 that will be it is it is coming out as 0, so there is no need of doing this one and is equal to 1695 into 0.475. So from here we are getting this L prime as this 396.6 kg, so we will be getting this V prime is equal to 1695 minus 396.6, that is coming out as 1298.4 kg. So, the mass of this overflow that is overflow that is we say V prime that is 1298.4 kg.

So, we can say this one that fractional fractional oil recovery, so in percentage if we say will be like this say V prime into YCV prime divided by so whatever the F prime we have into 100, so that is coming out as V prime is 1298.4 into YC prime is equal to say 0.115 divided by say 195 say into 100 percent. So it coming out as 76.6 percent, so here we are getting 76.6 percent and for that case we got 74.8 percent, so we can this one both are almost same and if we do this one very precisely means say if we do the calculation in a big graph paper like this, we will be getting the same, we will be getting the same fractional recovery.

So thank you and the next class we will be discussing about the multi stage of leaching operation.