

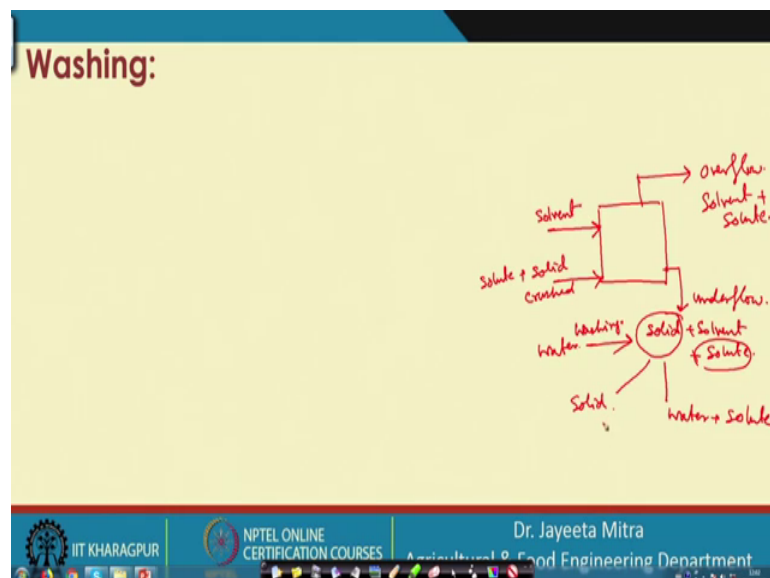
**Fundamentals of Food Process Engineering**  
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**Lecture – 54**  
**Leaching and Extraction (Contd.)**

Hello everyone. Welcome to the NPTEL online certification course on Fundamentals of Food Process Engineering. And we are discussing now Leaching and Extraction. We have a little discussed some part of leaching and extraction, the basic fundamental principle of leaching and extraction, the equilibrium of leaching, single stage and multistage leaching operation. We have done some problem on this and also we have discussed that the mass transfer rate of mass transfer equation in leaching.

Now, in today's class we will look some more you know, some more cases that are important in case of leaching and then we will move on to the extraction specifically the liquid liquid extraction process. And we do not have much time for elaborate discussion of this leaching and extraction. It is a really vast you know topic and in this particular course, we are just discussing the fundamental aspects. We are not going very deep into it may be you can refer other courses and books. And if you know in later stage, we may give some more elaboration on that but now, we will just give you the basic idea of what happens in case of the leaching and extraction process.

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So, after discussing the different stages and different operation of leaching and also discuss the equilibrium and the equilibrium conditions and the mass transfer rate of mass transfer then, next important thing in leaching is that washing of the slurry material. What happen you know; we are till now concerned about what the solute component we are extracting right. So, we have taken the material the solvent in such a way that, the solvent had a proper affinity towards the particular solute that we try to extract from the solid crush material or slurry material ok.

So, that was our main aim. So, that is why, we try to evaluate the over flow where the solute and solvent is there and then to separate the solute and solvent we apply other distillation or crystallization etcetera. But in some cases you know, the solute material is of important. And in that we actually try to separate one or more component from the solid material and then the pure material is of our requirement.

For example sometime during the cheese preparation, we want to remove the stress of the lactose then, that is being done by repeating washing treatment. So, in that washing actually, normally we try to take water if because, it is freely available and inexpensive. So, if the case can be can be solved by the; or washing can be solved can be done by water, it is very good and if it is not then we have to look for some other solvent that is specifically used for this kind of material.

So physically if you want to think the case is that, till now when we performed leaching, the solvent; solvent was coming and there was some solid material ok, solid may be crushed material mostly because. we want to increase the surface to volume ratio for all the cases so that, all the particle will come in intimate contact with the solvent and then we are getting here has an under flow the solid plus plus solvent plus solute component, some fraction of that and in the overflow we have the solvent plus solute.

Now, if the pure solid is our requirement what we need to do, we wash it with some solvent mostly we use water. So that, we want at the end that; the solid will be pure solid will get in the pure form or then we dry it to remove that that water and all the you know solute will come out of the solid material ok.

So then the then for washing this solute that is mixed with this solid is considered as an impurity ok. And that impurity is taken out by water and here the solid will be recovered. So, this may be actually moved. So therefore, washing is important, when the solid is of

important and the solid we want to extract in the pure form. So all the solute and solvent what is mixed with that we want to wash it and we get the pure solid material.

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**Washing:**

- ✓ Washing is similar to extraction.
- ✓ In washing inert material is required product and solvent is water.
- ✓ In multiple washing, the water content of the material is  $x_w$  (weight fraction) and a fraction of this,  $x$ , is impurity, and to this is added  $y x_w$  of wash water.
- ✓ The impurity, formerly contained in  $x_w$  of water is now in a mass  $(x_w + y x_w)$ ; its concentration  $x$  has fallen by the ratio of these volumes, that is to  $x \left[ \frac{x_w}{x_w + y x_w} \right]$

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So let us see now, in the washing how we approach It is similar to extraction similar in the sense in the extraction the solvent is used to take the solute out of the solid matrix and in the washing the whatever impurities of the solid residue remain in the solid that is extracted by some washing liquid that is normally water ok. In washing the inert material is required product; inert material is our required product that means our solid that we want to separate and the solvent here is water. Multiple washing, the water content of the material is  $X W$  so.

But happen that in 1 washing we may not fully you know take out the impurities that is mixed with the solid. So, in multiple washing, we wash repeatedly and the water content of the material let us say  $X W$ , that is the weight fraction and a fraction of this  $X$ , which is the impurity that is the solute or purity that we want to separate.

And to do this, what solvent we add, we add water of  $Y$  into  $X W$  amount ok. So, the impurity formally contained in  $X W$  amount of water because, initially the water content of the material was  $X W$ . So all the impurities where there in the  $X W$  ok.  $X X W$  of the water is now in the mass. So, once we try to wash it so then, the all the impurities will coming into the mass so it is coming into the  $X W$  plus  $Y$  into  $X W$  amount of water ok.

So  $X$   $W$  was already there in the sample or the solid material and  $Y$  into  $X$   $W$  that we have used for washing. So, it is concentration  $X$  has fallen by the ratio of these volumes that is now  $X$  ok.  $X$  into this will be  $X$   $W$  because, initially the fraction we have considered for  $X$   $W$ . So  $X$   $W$  by  $X$   $W$  plus  $Y$  into  $X$   $W$ .

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**Washing:**

after one washing:

$$x_1 = x \left[ \frac{x_w}{x_w(l + y)} \right] = x \left[ \frac{l}{l + y} \right]$$

after two washings:

$$x_2 = x_1 \left[ \frac{l}{l + y} \right] = x \left[ \frac{l}{l + y} \right]^2$$

and so after n washings:

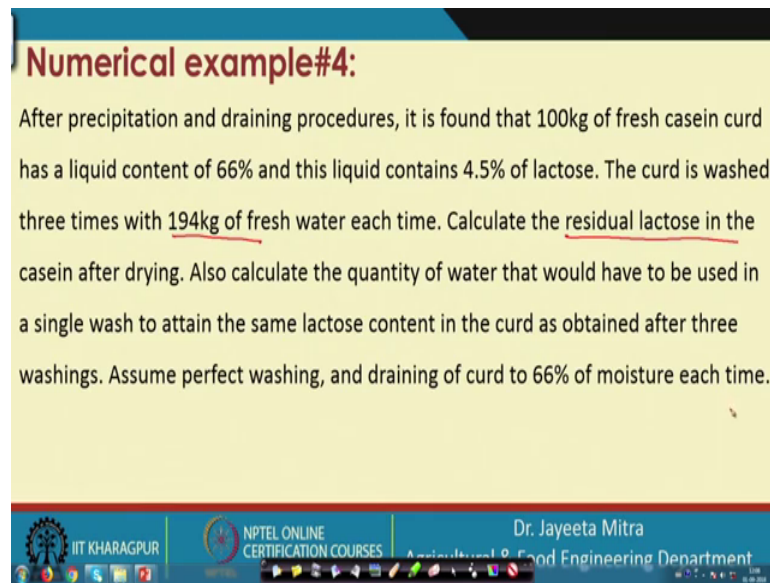
$$x_n = x \left[ \frac{l}{l + y} \right]^n$$

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So, now after 1 washing, when we perform one washing so what will remain that  $X$  1 the fraction that remain  $X$  1 that is  $X$  into  $X$   $W$  by  $X$   $W$  into 1 plus  $Y$  ok. So,  $X$   $W$  we can cancel it, so we are getting  $X$  into 1 by 1 plus  $Y$ . So,  $X$  was the fraction of impurity, so after 1 washing the level of impurity remain as this  $X$  1 that is  $X$  into 1 by 1 plus  $Y$  ok.

Now after two washing, this will become  $X$  2 into initial fraction was  $X$   $Y$ , so  $X$  1 into 1 by 1 plus  $Y$ . So,  $X$  1 is again  $X$  into 1 1 divided by 1 plus  $Y$ . So, finally, we are getting  $X$  into 1 by 1 plus  $Y$  square. This is for the 2 stage. Similarly, for the 3 stage, we will getting  $X$  3 that is equal to  $X$  into 1 by 1 plus  $Y$  to the power 2 and for  $n$  stage in the similar way, we are getting  $X$   $n$ , that is equal to  $X$  into 1 by 1 plus  $Y$  to the power  $n$  ok. So, this is how after each washing treatment, the impurity that remains ok. So, after one washing two washing  $n$ th washing this is the impurity remains or we can say the impurity concentration in the on the solution because, that is what we are calculating here.

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**Numerical example#4:**

After precipitation and draining procedures, it is found that 100kg of fresh casein curd has a liquid content of 66% and this liquid contains 4.5% of lactose. The curd is washed three times with 194kg of fresh water each time. Calculate the residual lactose in the casein after drying. Also calculate the quantity of water that would have to be used in a single wash to attain the same lactose content in the curd as obtained after three washings. Assume perfect washing, and draining of curd to 66% of moisture each time.

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So based on that, we will solve the problem now. And so, the problem is that after precipitation and draining procedure it is found that 100 kg of fresh casein curd has a liquid content of 66 percent and this liquid contain 4.5 percent of lactose ok, four point five percent of lactose.

Now, the curd is washed 3 times with 190 kg of fresh water each time. Calculate the residual lactose in the casein after drying ok. So, we want to actually remove lactose from the casein miscella. 4.5 percent was initially we try to wash it 3 times with 194 kg of fresh water. So, calculate the quantity of water that would have been would have to be used in a single wash to attain the same lactose content.

Now, there is a method that if suppose n times we are washing. So we can replace this with n times more water in one stage so, that can also be done So, assume the perfect washing and draining of curd to 66 percent of moisture each time. This was the condition given. Now let us see how we can solve this one ok.

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**Numerical example#solution#4:**

100kg of curd contain 66kg solution. The 66kg of solution contain 4.5% that is 3 kg of lactose.

In the first wash  $(194 + 66) = 260$ kg of solution contain 3kg lactose.

In 66 kg of solution lactose remaining there will be  $(66/260) \times 3 = 0.76$  kg.

After the second wash the lactose remaining will be  $(66/260) \times 0.76 = 0.19$  kg.

After the third wash the lactose remaining will be  $(66/260) \times 0.19 = 0.048$  kg.

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So, 100 kg of the curd contain 60 kg, 60 kg of the solution, 100 kg of the curd contain 60 kg of solution and the 66 kg, 66 kg solution, so 66 kg solution contains 4.5 percent that is 3 kg of lactose. So, in the first wash, we had 194 for washing water and this much solution were there ok. So, solution means this much kg of water in that the lactose is there. So, total 260 kg of the solution that contain 3 kg of lactose after 1 wash.

So, in 66 kg of solution lactose remaining, there will be 66 divided by 260 into 3 because, initially it was 3 kg and we have seen that the percentage X will be multiplied each time with this ratio, so we are getting 0.76 kg. Now next time with 0.76 we apply this much ratio. So after this second wash, the lactose remaining will be 66 by 260 into 0.76 ok, that is that remain in the last stage. So, it is 0.19.

Similarly, after the third wash, the lactose remaining will be 66 into 260 into 0.19, so we are getting 0.048 kg.

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**Numerical example#solution#4:**

Or, after three washings lactose remaining will be  $3 \times (66/260)^3 = 0.048$  kg.

So, after washing and drying 0.048kg of lactose will remain with 34kg dry casein so that lactose content of the product =  $0.048/34.05 = 0.14\%$

and total wash water =  $3 \times 194 = 582$ kg

To reduce the impurity content to 0.048 kg in one wash would require x kg of water, where

$(3 \times 66)/(x + 66) = 0.048$ kg;  $x = 4060$  kg. so the total wash water = 4060kg.

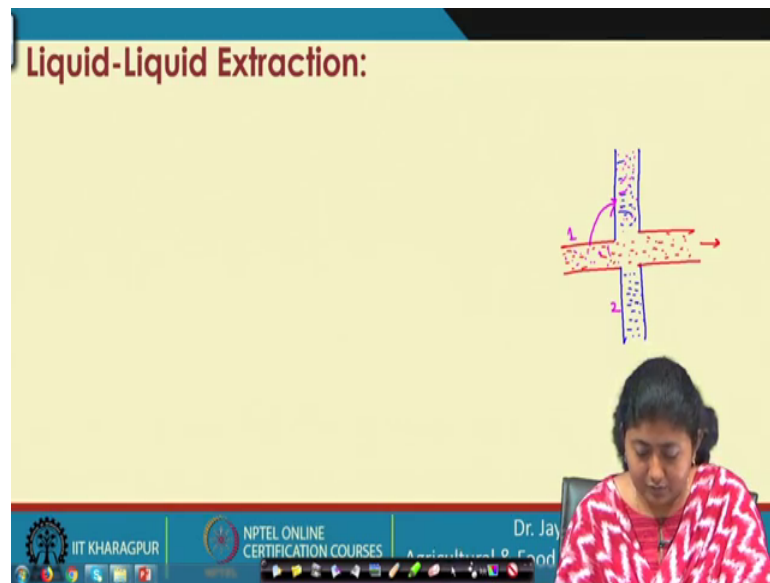
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So, after the 3 washing the lactose remaining will be 3 into 66 into 260 to the power 3. So, this is by the empirical equation that they have given, so 3 into 66 into 260 to the power cube that is 0.048 ok. And simply also by the other method also we are also getting the same thing. If we just replace each time fraction with this 66 by 260 ratio of the water divided by the total water.

So, we are getting the same value that is 0.048 kg. Now, after washing and after washing and drying 0.048 kg of lactose will remain with 34 kg dry casein. So, that the lactose content of the product will be 0.048 by 34.05. So, 0.14 percent so because, because 66 was the solution, so from the 134 kg was the dry casein.

Now, the total wash water was 582 because, 3 into 194. So, to reduce the impurity content to 0.048 kg in one wash would require X kg of the water where 3 into 66 by X plus 66, that is equal to 0.048. So, we are getting 4060 kg. So, the total wash water is 4060 ok. So, this will require if we want to achieve this much impurity level in one stage washing.

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So, next we will start liquid-liquid extraction. So, far we have mostly discussed the leaching that is the solid liquid extraction process. And we have discussed it is equilibrium, multistage process, operation mass transfer rate and few equations. And finally, the washing of the washing method for extracting the solid component, when the solid component in pure form is required; that is coming from the underflow.

Now, next we will discuss the liquid-liquid extraction So, in the liquid-liquid extraction, the main use of this is there in some bio separation process then, some flavour extraction and oil extraction or when we want to separate the antibiotics all this things from one solution to the other. So, in that case, we use this one liquid-liquid extraction and here like in the solid liquid extraction, the solid and liquid though both came into very intimate contact. Here, 2 liquid phases are coming into contact and a third component that has affinity higher than the second liquid from the first so it will going to going to the second one.

For example, if we try to visualize this, suppose 1 fluid stream is coming which is having, which is having some you know solid particle. And then the then the other stream, the other stream is getting mix with it other stream is getting mix with it which is having the pure solvent may be there will be no tresses of there may some tresses of the solute or may be ok.



So, then what will happen that, what will happen that, this the first solvent or liquid that has and had that solute earlier, will come out from the system and all the solute all the solute will now come to the second liquid of the second solvent. Because, this solute has higher affinity to the second liquid or the second solvent that we have use to extract that from the first phase or the first liquid phase ok.

So, phase 1 and 2, these two liquid phase are come into direct contact and the solute component has been taken by this, solute component has been taken by the solvent, second solvent and the first solvent will go out. So, the concentration of the solute will increase in the second solvent and while it is decrease in the first one and the equilibrium in these two when it will reach, so that time the process will be stop, because of achieving the equilibrium condition. So, liquid-liquid extraction again the transfer is based on the molecular transfer.

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**Liquid-Liquid Extraction:**

In liquid-liquid extraction the extraction takes place from one liquid medium to another.

- ✓ Feed contains the desired solute.
- ✓ Mass transfer takes place.
- ✓ The raffinate is the residual feed solution, may contain little solute.

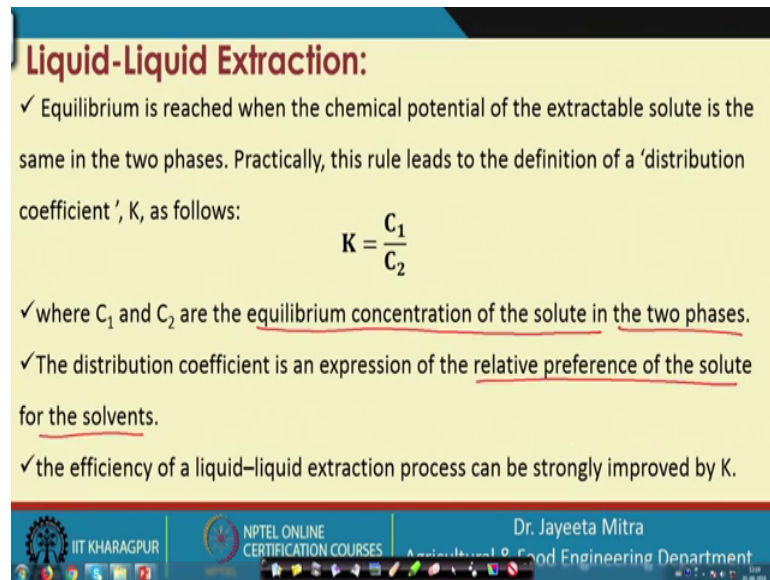
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So, in liquid liquid extraction, the extraction takes place from one liquid medium to the other as we have mentioned So, we have a feed we have a solvent there is an extractor.

Now, the raffinate we call when the solute is come out from the feed solvent and extract we call when the solute is coming with the solvent and the solvent is going out. So, the extract is this one and the raffinate is this one. So, this term is practically used for a liquid liquid extraction only.

Here the feed contain the desired solute, mass transfer takes place, because of these 2 having the potential, chemical potential difference for that for that particular solute. So, through their interface layer or contact layer the transfer of mass will takes place. The raffinate is the residual feed solution they contain the little solute also.

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**Liquid-Liquid Extraction:**

- ✓ Equilibrium is reached when the chemical potential of the extractable solute is the same in the two phases. Practically, this rule leads to the definition of a 'distribution coefficient', K, as follows:

$$K = \frac{C_1}{C_2}$$

- ✓ where  $C_1$  and  $C_2$  are the equilibrium concentration of the solute in the two phases.
- ✓ The distribution coefficient is an expression of the relative preference of the solute for the solvents.
- ✓ the efficiency of a liquid-liquid extraction process can be strongly improved by K.

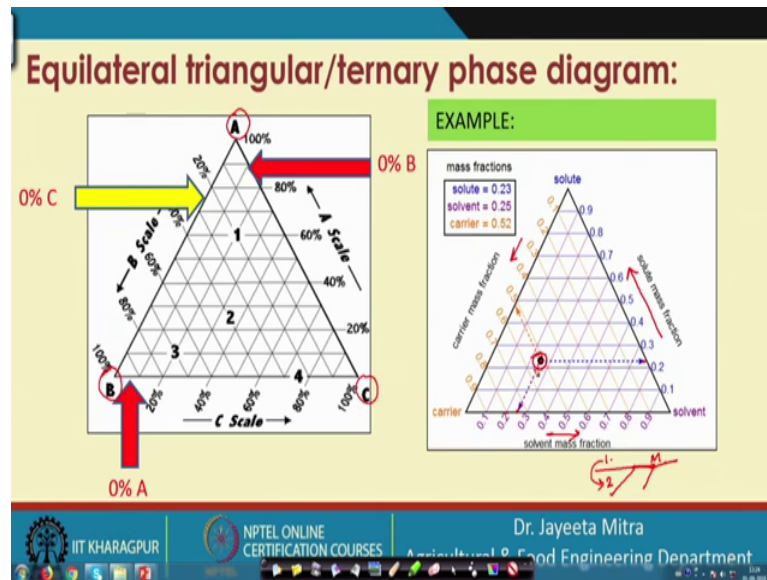
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So as we need the equilibrium condition in leaching, we need it we have to observe that equilibrium condition in the solvent extraction or liquid-liquid extraction also. So, equilibrium is reached when the chemical potential of the extractable solute is the same in the 2 phases. Practically, these 2 leads to the definition of a distribution coefficient K, that is equal to C 1 by C2. So, distribution coefficient is expressed as this and C1 and C2 are the equilibrium concentration of the solute in the two phases because, 2 liquid phases are coming into contact so that extract and raffinate these two are going out. So, actually these are the concentration of this extract and raffinate phase.

The distribution coefficient is an expression of the relative preference of the solute for the solvent ok. So, ideally this will be uniform when the equilibrium condition is arrived, but because we need to know the relative preference because, each solute is having in one solvent and the affinity to that solvent is higher, than the other solvent then the extraction would not be possible. So, that we have to see.

Now, the efficiency of a liquid liquid extraction process can be strongly improved by K. So, if the distribution coefficient increase, so that liquid-liquid extraction efficiency will also increase.

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Now, as we have shown you how to plot the equilibrium conditions in case of leaching operation there we have taken in a rectangular plot and in the Y axis we plotted  $n$ , that is the inert material divided by the solute kg of the solute plus kg of the solvent, with that we have plotted  $Y_A$  and  $X_A$  that is the solute fraction in the feed in the slurry stream and in the extract or in the in those cases for the leaching.

Similarly, for the liquid-liquid extraction also we can develop the equilibrium plots. And these equilibrium plots are of many types ok. So, sometime we may refer the equilateral triangular-triangular plot or rectangular plots are also there, even these liquids that that 2 phase liquids that we are using to separate some compounds some solute or some you know some oil some flavor compound etcetera.

So, sometime those 2 liquid that we are using those are partially visible also. So, there are many cases here we will discuss 1 or 2 of them. So, equilateral triangular or ternary phase diagram ok. So, we need to have 3 component because we have 2 solvent and one is the component that we want to separate.

So, you can see this equilateral triangular or ternary phase diagram for leaching were. ok. Where, you can consider a that is the solute that we want to extract and B that is the solvent 1 solvent and C is the other one. So, scale of C moving from this side to this side ok, from 0 percent to 100 percent and scale of A varies from here to here 0 to 100 percent and scale of B varies from here to here ok.

So, if this is one approach there are many approach of solving this. So, let us say one case we want to have, so how this 3 can be separated. Initially we will get a mixture. Suppose we are having 2 stream, stream 1, stream 1; we are having and stream 2 we are having.

So, when they will get mixed, so they will reach eventually on equilibrium stage ok, that is even equilibrium stage let us signifies by M ok. And when this equilibrium stage is coming, so we will get phase 1 and phase 2 separated, so the solute component of the phase 1 has now reached to phase 2, but this point when we say that this is equilibrium. That means, that equilibrium should be visible by measuring the concentration of that solute in both the stream. And that should come equal or in the ideal case, it is coming equal we may not get equal, but we can calculate that from those charts.

So, then the main thing is we need to achieve some equilibrium position ok. So, in this example, let us say we have a point, we have ternary phase diagram and here we have a point ok. Now in this point what will be the concentration that we want to measure? Now the concentration suppose we define A as the A as the you know 1 1 solute component that we want to separate and B or C as 1 solvent.

So, in 1 stream the concentration of A if it is fixed, so the other concentration we can fix So, we can see that, we consider this point we can see that solute varies from 0 to 1 in this direction. Solvent mass fraction varies from this left to right in this session and the solute solvent fraction. So, this is first one is the carrier, so this is 1 1 solvent that is the carrier 1 that is carrying this solute. This is the solvent and this is the solute solid mass fraction right. So, at this point, we can have the line just parallel to this already having this ternary line. So, we are getting a little higher than 0.5, so let us say 0.52 that is, the mass fraction of the carrier ok.

And now, if we draw this line on the solvent side, so we are getting that around 0.25 that will be the concentration of the solvent. And similarly, if we move towards the solute

side, we are getting also around 0.23. So, this is how all the 3 fraction can be find out at a particular condition.

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**Key points to remember:**

- ✓ Each vertices of triangle represents pure component.
- ✓ Each base line indicates the composition of a binary mixture.
- ✓ In each base line, the composition of a pure component located opposite to that vertex is zero.
- ✓ In liquid- liquid extraction process, the three vertices indicate **SOLUTE, CARRIER** and **SOLVENT**.
- ✓ In LLE, ternary diagram has two regions- a) Single liquid region.  
b) Two liquid phase region.

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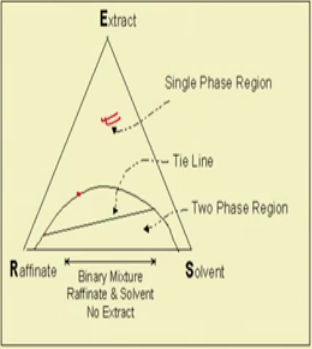
So, in the leaching sorry in the solvent extraction or liquid-liquid extraction process we need to remember few things. First is each variety of triangle represent pure component, each vertices of the triangle you present the pure component. And each base line indicate the composition of a binary mixture, each base line having the composition of the binary mixture. So, in base line, the composition of pure component located opposite to that vertex is 0. And in liquid-liquid extraction process, the 3 vertices indicates the solute carrier and solvent, that we have seen just now in that three plot.

And in liquid-liquid extraction, the ternary diagram has two region; one is the single liquid region and the 2 liquid phase region; so this we actually when we have some partially visible solvent.

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**Two regions in ternary diagram:**

- ✓ A single liquid region, where all 3 components are dissolved in a single liquid phase.
- ✓ A two liquid phase region, where mixture composition splits into two immiscible liquids. For LLE the mixture needs to be in 2 phase region.
- ✓ The two regions are divided by miscibility boundary or liquid-liquid equilibrium line.



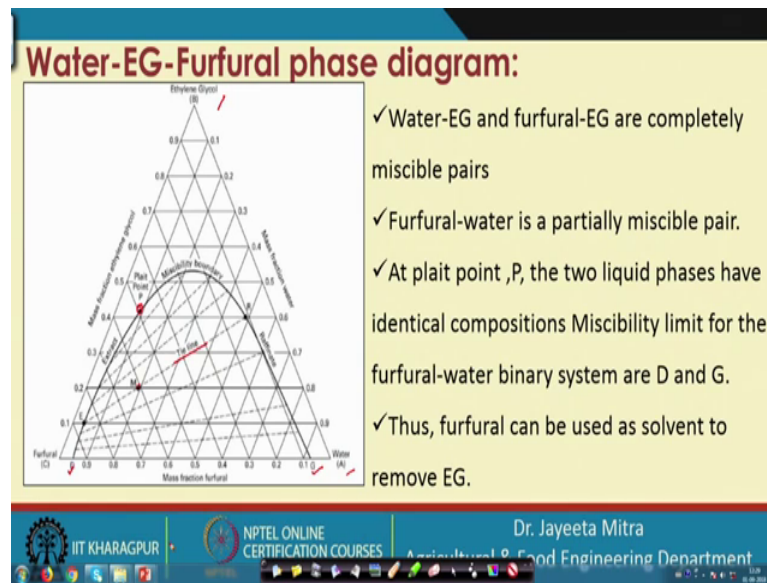
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So, here we can see in this the diagram that, we have we have extract that we want to separate we have raffinate and we have solvent ok. So, this is the solvent, this is raffinate that is after the taking out that solute the stream is called raffinate and here it is the extract. So, a single liquid region where, all 3 components are dissolve in a single liquid phase that we can have from here and that two liquid phase region.

So, this is the single-single phase region where, only the extract component will be there and there is a two phase region where the solvent and raffinate will be getting partially mixed. The 2 liquid phase region where mixture composition split into two invisible liquid, for liquid-liquid extraction, the mixture needs to be in 2 phase region So here it is 2 phase region and this lines are called the tie lines ok.

And there will be 1 point that is called the plate point that actually having a concentration of both the solvent similar or both are coexist at that condition. So, the two regions are divided by visibility boundary or liquid-liquid equilibrium line, so this is the visibility boundary or liquid-liquid equilibrium line.

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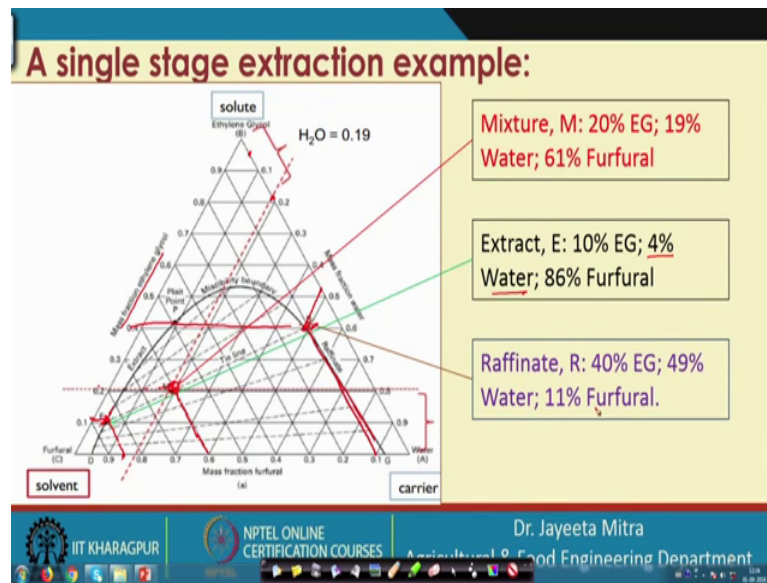


So, now, this is again 1 example that, we solve in this across that visibility boundary is given and the mass fraction of furfural is here, ethylene glycol that we want to separate from water using this ok. So, there is the water and ethylene glycol and furfural ethylene glycol are completely miscible pair. Water and ethylene glycol and furfural and ethylene glycol now, furfural and water is partially visible pair ok.

So, water and furfural is partially miscible and there is a miscibility boundary So, this is called the plate point. At plate point P, the 2 liquid phase have identical composition ok. So, miscibility limit for the furfural water binary systems are D and G. So, here it is D and G. So, that is from this composition to this composition, it can be mixed among themselves.

And, we can observe this are the tie line, the tie line so after this combination; that means, the water ethylene glycol is mixed with the furfural. So, 1 point will be there that that is the mixing point and from that point we need to find that what is the concentration of all other all other you know raffinate and extract sessions ok.

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So, in this particular diagram, what we can see that we denoted this point M that the mixture point ok.

So, in this point mixture we need to find the percentage of ethylene glycol, then water and then the furfural. So, we will have a line straight from this point M and that will that will that will touch this line of the mass fraction of water at some points here, which is around 19 percent because, it is moving from this direction 0 to this so we are getting at nineteen percent.

Then, we will look for the mass fraction of ethylene glycol. So, this is the ethylene glycol mass fraction. So, we will go in this left side and find that; what is the fraction of the point. So, we are getting that 20 percent of ethylene glycol, 19 percent water and the percentage of the furfural now we need to find.

So, from the first furfural we will just straight away coming this side ok. So, around 61 percent of that we will get ok. So, we will we get furfural here we get, mass fraction of the ethylene glycol here and we will get here the water ok. Now, what we need to do is, from the mixture we need to find; what is the composition of the raffinate and what is the composition of the extract.

So, for raffinate and extract this passing through M, we will need to have the tie line ok. So, here is the tie line is there this is E and this is the R. So, this is the raffinate and this is



the extract. So, initially ethylene glycol was mixed with the water now it will come to the furfural. So, that is why it is extract ok.

So, then what we will get that if, if at this extract point we want to find that what is the percentage of the ethylene glycol, so we will we can get here that is 10 percent, 10 percent ethylene glycol we are getting. And now, what is the water in this because, some we cannot completely separate so, from here we will look for that how much percentage. So, if we will get somewhere here we will end up, so that is around 4 percent water, around 4 percent water we will get. And from this line, extract line if we come here, so somewhere here we are reaching, so 80 percent furfural will be there.

Now coming to the raffinate part, so for the raffinate part if you try to read the equilibrium diagram, so for the raffinate we will have the tie line from the mixture to the raffinate side and at this point. So, we will have straight away, ethylene glycol and that is 40 percent and we have water as around 40 48 or 49 percent. And similarly, we have the we have from this point from this point, we have the we have around this is our vapor zone, so somewhere here will have, so around 1 percent of the furfural.

So, this is how the equilibrium diagram in case of the liquid liquid extraction can be read. We are not going to give you any equations there because. These equations are almost similar that we have developed in the leaching. We have 2 streams that is coming in to there are different fraction of that particular solute component and then those 2 are getting mixed and extract and raffinate are coming.

So, all they are almost similar to that but, we want to give you an idea of how to read those how to read this you know, equilibrium charts if it is a miscible 2 2 solvents are partially miscible or if they are not then we can have simply equilateral triangular diagram or ternary phase diagram. And we are not going to very detail discussion of this but, this will give you an idea that how the separation is take taking place and what is the fraction of the component that is going into each phases ok.

So, we will stop here. And we will discuss the rest of the thing in the next class.

Thank you.