### Fundamentals of Food Process Engineering Prof. Jayeeta Mitra Department of Agriculture and Food Engineering Indian Institute of Technology, Kharagpur

## Lecture - 53 Leaching and Extraction ( Contd. )

Hello everyone, welcome to the NPTEL online certification course on Fundamentals of Food Process Engineering. We are in the chapter 11 now and in the last two classes we have discussed about the Leaching and Extraction process. Leaching and extraction is a separation technique similar to that adsorption or distillation or crystallization.

However, the difference is that this has certain advantage over the distillation or crystallization evaporation those kind of methods. Because, here we do not need excessive high temperature, we can separate the solute or particular solute component which is in the in component mixture by some other phase, may be by some other liquid phase. And, we can separate the particular component without application of heat ok.

So, for heat sensitive material leaching and extraction is very important and it is also important when we have very dilute solution of a particular component that we want to separate. And, the in food there are many application of leaching and extraction we can find some of them we have discussed. Leaching is basically when the solid component comes into contact with a solvent and a particular solute from the solid is being separated because, that solute has a particular affinity towards the solvent ok. But, when instead of solid we use two liquid phase and one component from one liquid will be extracted by the other liquid then we call it extraction or solvent extraction ok.

So, both the method either solid liquid extraction or liquid extraction these two are very important in food processing. We have seen that green tea extraction, if you want to develop green tea from the green tea leaf powder or granules or if the caffeine if the coffee beans are used for extraction of the caffeine. So, all this are application of leaching and extraction is used when some let us say some components of the oil or flavor that we want to separate by utilizing a some other solvent and then we call it extraction. So, basically for oil we definitely use that solvent extraction technique and all this two cases we need to know few conditions and few parameters.

So, some of them we have discussed in our precious two classes; one is the equilibrium condition because these two separation techniques are actually the molecular transfer techniques. The solute molecule is moving from one phase to the other because, of the difference in the chemical potential or difference in the concentration. So, this molecular transfer will occur unless there an equilibrium will be established right. So, when the equilibrium will be established then this process the transfer of the solute from one phase to the other will stop.

So, that equilibrium we have discussed the equilibrium for single stage leaching operation in our last class. Today we will solve one problem on that and also we have seen the multistage leaching, where the single stage operation is repeated the same way we can perform for the multistage. And, we can assume that the transfer the rate of transfer will be same the rate of transfer of the mass will be same in each phase and eventually the equilibrium will be attained. So, basically by equilibrium we want to mean that the chemical potential of the solid phase from where we are extracting the solute or the slurry that we call it and the extract the solvent that we are using.

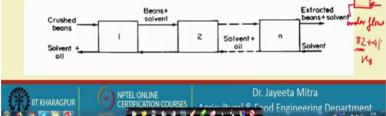
So, in this two stream when the chemical potential of a particular solute will be similar, then the equilibrium will be achieved. Now, this is an ideal case it may not happen that ideally, if for a finite period of time the contact between these slurry and the solvent is taking place. So, may not be the ideal situation we can find so, for that we have to make this contact for a longer time for a infinitely long time so, that this equilibrium will be reached. So, in ideal case we may get some deviation so, the concentration or the fraction of the solute in the slurry and solvent and in the extract may be different ok.

So, we have seen that we have also seen the how to draw the curve between the inert fraction, in the solute plus solvent stream and with the solute fraction in a solute and solvent stream; how the equilibrium diagram can be drawn for the leaching. And, also we have seen that how the operating line and the equilibrium line can be plotted for a single stage operation. Now, we will solve a problem and this problem is based on the multistage in the previous class we have discussed the phenomena that happened in a multistage although it is similar to the single stage only and it is a counter current flow.

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# Numerical example#2:

Oil is to be extracted from soya beans in a counter current, stage contact, extraction apparatus, using hexane. If the initial oil content of the beans is 18%, the final extract solution is to contain 40% of oil, and if 90% of the total oil is to be extracted, calculate the number of contact stages that are necessary. Assume that the oil is extracted from the beans in the first mixer, that equilibrium is reached in each stage, and that the crushed bean solids in the underflow retain in addition half their weight of solution, after each settling stage.



So, in a counter current leaching from the one side the crushed bean, this is our the solid mixture that is entered ok. And, from the crushed bean we want to extract the soya oil that we want to extract and there is a solvent that is entering in to the multi stage leaching apparatus from the opposite side ok so, this is the counter current operation. Now, let us see what is the question and then step by step we will try to solve it.

So, the question is oil is to be extracted from soya bean in a counter current, stage contact, extraction apparatus ok. So, multistage and there are different stages are there in every stage the beans, the solid material and the solvent are coming in close contact. And, initial content of the initial oil content of the bean is 18 percent ok. The final extract solution is to contain 40 percent of oil ok so, the extract that we are getting at the end that is the solvent plus oil. So, the that should contain 40 percent of oil and 90 percent of total oil is to be extracted; that means, the bean has some amount of oil that is 18 percent.

And, of that 90 percent should be oil should be extracted. Calculate the number of contact stages that are necessary to separate this 90 percent of the initial sample. Assume that the oil is extracted from the beans in the first mixture, that equilibrium is reached in each stage and that the crushed bean solid in the under flow retain in addition half their weight of the solution after each settling stage. So, we have seen while in the single stage or we have seen that if this is your extraction ok vessel, let us say for a single stage and when your solvent is coming into it and the solid is also entering.

So, there will be under there will be over flow which is having your solvent plus solute that we want to extract or may be solvent plus oil that you want to extract. And, the slurry will be your under flow ok, slurry will be your under flow. So, here it says that so, crushed bean solid in the under flow retain in addition half their weight of solution after each settling stage. That means after one stage let us say that beans is having 18 percent 18 percent oil.

So, if we take total 100 percent material is coming into the system so, in a first stage 18 percent is the oil. So, 82 percent will be the solid beans solid material. So, in the underflow this 82 will go along with that half the solution will be going. So, plus 41 this much kg of the material will have solvent plus some solute because, not all the solute will be extracted 90 percent will be extracted. So, this is how we can approach the problem and let see we have solved it for some stages.

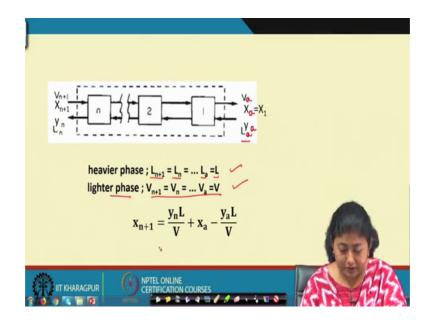
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Multiple stage	e counter cu	rrent leaching:
Material balance:	$V_{n+1} + L_a =$	$V_a + L_n$
	$V_{n+1}x_{n+1} + L_ay_a$	$= V_a x_a + L_n y_n$
✓Eliminating $V_{n+1}$ bet	ween these equation $(L_n - L_a + V_a)x_{n+1} = V_a$	
	$x_{n+1} = y_n \Big[ \frac{L_n}{L_n - L_a} +$	$\overline{V_a} \bigg] + [\frac{V_a x_a - L_a y_a}{L_n - L_a + V_a}]$
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And, before going into that just I will once again remained you the equation of material balance and component balance which is used in the multistage. So, in the multistage as this is the multistage diagram and this is the V n plus 1, that is the solvent entering into the n th stage. And, L a that is the solid material of from which we need to extract a particular component or solute or oil that the rate is L a ok. This will be equal to V a plus L n, where V a is the solvent or the extract that we are getting from here and L n is the slurry from the n th stage ok.

Similarly, when we do the component balance in that so, V n plus 1 into x n plus 1 that is the concentration of the oil or solute, the particular component that we want to separate that in the solvent itself. And, similarly x a denote that the component fraction in the extraction that is coming out from the first stage. And L a y a so y a is the fraction of that solute component in the feed stream and y n is the fraction of the that solute components that coming from the n th stage ok.

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And, from this two we can eliminate V n plus 1 and then we develop this equation just taking the n plus 1 from here and then we put in the balance equation. So, L n plus V a minus L a that into x n plus 1 that is equal to V a x a plus L n y n minus L a y a. And, from here we can get the x n plus 1 as this y n into L n by L n minus L a plus V a plus V a x a minus L a y a divided by L n minus L a plus V a. So, this is the material balance and over all mass balance or material balance and the component balance on the solute so, this we will use now to solve the problem.

So, we can see that V n plus 1 is the solvent entry in the n th stage x n plus 1 the concentration the fraction of that solute in the stream. L n Y n is the slurry that comes out from the n th stage, X 0 or X a we can we can take this as because we have used this notation L a Y a X a and V a right. So, heavier phase heavier phase means the feed slurry that that enter. So, that is we have assumed same because, the is the condition given that the rate of transfer will be same in each stage right. So, L n plus 1 that is equal to L n that

equal to L a that equal to L so, this that the stream that is entering in each consecutive stages are same.

Similarly, the lighter phase so, V n plus 1 that is equal to V n that is equal to V a eventually V. So, this stream is having also same heavier phase and lighter phase, lighter phase means the solvent, heavier phase means the feed stream or the feed slurry. And, we have got this applying this two in the previous equation x n plus 1 equal to y n L by V plus x a minus y a L by V. So, we had first the material balance equation and component balance equation from where we got some expression of x n plus 1. Now, considering these two approach we are simplifying the equation of x n plus 1.

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Numerical solution 2:
Overall mass balance:
100kg raw material with 18% oil, i.e., 82kg bean solids and 18kg oil. In the final
underflow, 82 kg bean solids will retain 41 kg of solution, the solution will contain
10% of the initial oil in the beans, that is, 1.8kg so that there will be (18 - 1.8) =
16.2kg of oil in the final overflow, AI-18 (39.2) kg.
Extract contains $(16.2 \times 60/40) = 24.3$ kg of solvent
Total volume of final overflow = 16.2 + 24.3 = 40.5kg
Total solvent entering = (39.2 + 24.3) = 63.5kg
Note that the solution passing as overflow between the stages is the same weight
as the solvent entering the whole system, i.e. 63.5kg
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So, you can see now we have mentioned that let us say 100 kg basis, if we start 100 kg raw material that is raw soya bean crush material we have taken, in that 18 percent oil was there. So, 82 kg bean solid was there right and 18 kg oil. Now, in the final underflow so, what will be going there some oil will be extracted, but most of the all the solid will be going in the underflow. So, in the underflow 82 kg bean solid will be there and it was mentioned that half of that amount of this bean solid will be the solution part.

Because, we have discussed this that during leaching we cannot get the dry solution initially we will get two section; under flow and over flow. Over flow will have the solvent and the solute in it and the under flow will have all the solid slurry material and some solvent. And, that solvent will have some solute and the concentration or chemical potential of the solute in that under flow stream and the over flow if it these two are equal then only the equilibrium condition arise ok. So, that stream the underflow stream will have half of the weight of the miscella will be the solution so, 41 kg of solution. Now, the solution will contain 10 percent of the initial oil in the bean because, it was mentioned that in the question that the 90 percent of the oil was extracted right.

So, 10 percent means 10 percent of this 18 kg so, 1.8 kg. So, that 18 minus 1.8 this has gone to the over flow right. So, 18 minus 1.8 so, 16.2 kg of oil in the final over flow right. Now, it also mentioned that this much that is that oil is coming into the solvent, but it will have 40 percent of the solvent because, solvent is having 40 percent concentration of the oil in the extract. So, the extract contain 60 percent solvent and 40 percent solute so, if 40 percent is equal to the 16.2 kg, what will be the 60 percent? So, that is 16.2 into 60 by 40 so, we are getting 24.3 kg of solvent. Now, the total volume of final over flow so, over flow will have the solvent plus the oil in it so, 16.2 plus 24.3 so, 40.5 kg right.

Now, the total solvent entering that we need to find. So, if you remember we had 41 kg solution and in that solution we had 1.8 kg of the oil material. So, what was the solvent? So, 41 minus 1.8 so, we are getting 39.2 so, that 39.2 kg is the total solvent that has gone to the underflow and 24.3 that has gone to the over flow so, total solvent was 63.5 kg. Now, note that the solution passing as overflow between the stages is the same weight as the solvent entering the whole system. So, the solution passing as the overflow between the stages is the same that has entered into the system so, that is 63.5. Now, this we have to see in all the stages what is happening so, this is the overall balance.

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MASS	BALACE
Basis: 100kg beans	Basis: 100kg beans
Mass in (kg)	Mass out (kg)
Underflow	Underflow
Raw beans = 100	Extracted beans +solution= 123
Bean solids = 82 🗸	Bean solids = 82;Oil = 1.8;solvent= 39.2
Oil = 18 🗸	Overflow
Overflow	Total extract = 40.5;solvent =24.3
Solvent = 63.5	oil = 16.2
Total = <u>163</u> .5 🗸	Total = 163.5
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So, 100 kg in the underflow we are getting beans 82 we are getting bean solid 82, oil 18, overflow solvent is 63.5 so, total is 163.5. Now, then from the 100 kg basis 100 kg bean underflow that will be extracted bean plus solution ok. So, that is 82 plus 1.8 plus solvent that is 39.2.

So, total we are getting 123 as an underflow. What will be the overflow? So, overflow will be the total extract that is 40.5 in that solvent is this 24.3 and oil is 16.2. So, total we are getting 163.5 so, this plus this so, we are getting 163.5; so this total remains same in stage 1 ok.

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	Analysis of stage 1
	Oil concentration in uderflow = product concentration = 0.4.
	It is an equilibrium stage, so oil concentration in underflow equals oil concentration
	in overflow. Let $X_2$ represent the concentration of oil in the overflow from stage 2
	passing in to stage 1. Then oil entering stage 1 equals oil leaving stage 1.
	Therefore balance on oil:
	63.5X <sub>2</sub> + 18 = 41 x 0.4 + 40.5 x 0.4; X <sub>2</sub> = 0.23; X <sub>2</sub> = Y <sub>2</sub> = 0.23
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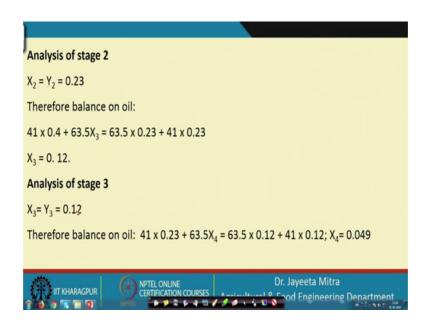
So, analysis of stage 1 is that the oil concentration in the underflow that is the product concentration that comes 0.4 40 percent was there and it is an equilibrium stage. So, oil concentration in the underflow equals the oil concentration in the overflow because, all stage will be in the equilibrium. So, let X 2 represent the concentration of oil in the over flow from stage 2, stage 1 we have calculated and we have got that as equilibrium concentration in the product and the underflow is 0.4. So, stage 2 X 2 represent the concentration of oil in the over flow from stage 1 equals the oil concentration of the stage 1. Then oil entering stage 1 equals the oil leaving stage 2 ok.

So, the oil entering here will be stage 2 from the stage 1 it is coming to the stage 2. So, the oil entering at stage 1 oil entering at stage 1 equals the oil leaving at stage 2. So, since the solvent is coming from the back side ok so, it is actually the oil which is leaving at stage 2 that is coming into stage 1 like that. So, therefore the balance we can calculate so, 63.5 that is the solvent which is having X 2 concentration plus 18 that is the oil that will be equal to 41 into 0.4 plus 40 into 40.5 into 0.4.

So, this is the underflow and the overflow and this is the initial stream so, initial stream has X 2 if we assume and that this 18 was in the field stream. So, X 2 we are getting 0.23 ok, X 2 is 0.23. Now, since X 2 will be equal to Y 2 because, the concentration in the equilibrium will be same in the underflow and the overflow ok. So, X 2 will be equal to

Y 2 so, we are getting 0.23 because, this will now enter in the this X 2 that that remains in the in that stream that will now enter into the next stage.

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So, similarly in the stage 2, were we have taken that X 2 equal to X 3 that is 0.23. So, this 41 into 0.4 plus this 63.5 into X 3 now. So, that will be equal to 63.5 into 0.23 plus 41 into 0.23. So, eventually we are getting X 3 as 0.12 and this analysis will go on we will take X 3 now, equal to Y 3 and that is equal to 0.12. So, if you just go back to the initial diagram. So, here we can see that then when in the in 1 stage, when in 1 stage the solvent is entering and the extract that that is coming out the overflow that is coming out that is entering as a solvent in the next ok. And, the extract that is coming out that is entering as a solvent in the next one.

So, like that in the first stage which is if you consider the first stage so, the exit of the second stage is entering the overflow of the second stage is entering at the solvent in the first stage right. And, eventually the concentration will change in a way so, X 1 that we are calculating so, in the equilibrium stage this will be same with the Y 1 ok. Similarly, the X 2 that stage will consider that will similar to the Y 2 and so on. So, we will one after the other the stage will count calculate so, finally, in the stage 3 X 3 equal to Y 3 0.12 and then we will do the balance. So, 41 into 0.23 plus 63.5 into X 4 that will be equal to 63.5 into 0.12 plus 41 into 0.12 so, we are getting X 4 as 0.049 like that. So, X 4 that means, in the 4th stage we are getting this.

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Analysis of stage 4
X <sub>4</sub> = Y <sub>4</sub> = 0.049
Therefore balance on oil:
41 x 0.12 + 63.5 X <sub>5</sub> = 63.5 x 0.049 + 41 x 0.049,
X <sub>5</sub> = 0.00315.
The required terminal condition is that the underflow from the final n <sup>th</sup> stage will
have less than 1.8 kg of oil, that is, that $X_n$ is less than 1.8/41 = 0.044.
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And, analysis of stage 4 finally, again X 4 equal to Y 4 because of the equilibrium condition so, 0.049. And therefore, the balance on the oil will be again we will calculate in the same way; that is 41 into 0.12 plus 63.5 into X 5 that is equal to 63.5 into 0.049 plus 41 into 0.049. So, we are getting this much 0.00315. Now, the required terminal condition is that the underflow from the final n th stage will have less than 1.8 kg of the oil because, 90 percent extraction should be done so, less than 1.8 kg.

Now, that X n if we try to calculate in the terms of fraction; so, X n is less than how much? X n that the fraction we want to calculate that is 1.8 out of 41 ok. So, the stream has 41 kg of the solvent in that 1.8 was there. So, at least lesser than this fraction we need to find out. So, 1.8 by 41 is 0.044 and here we are getting 0.00315 so, after 5th stage we are almost achieve the condition that 90 percent extraction of the oil has been done from the sample ok. So, this is how you can design when you want to setup any extraction solvent extraction or for example, you want to separate a leaching setup for that you have to think about what is your sample size and what is the flow rate.

And, how much oil content in the initial sample is having and you want to separate what amount of or what fraction of oil or solute you want to separate. So, based on that you have to design that how many stages are required ok. And also in the yesterday's class we have discussed the efficiency calculation that is by the Murphree efficiency; which based on what is the extraction can be achieved in the equilibrium condition. And, if equilibrium is not achieved so, how far we can have that concentration and the ratio of those two things we can use for finding the efficiency of each stage.

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Mass transfer in leaching operation:
✓ the equation for mass transfer may be written as:
$\frac{\mathrm{d}M}{\mathrm{d}M} = \frac{\mathrm{k}'\mathrm{A}(\mathrm{C}_{\mathrm{S}}-\mathrm{C})}{\mathrm{d}M}$
dt b
where: A is the area of the solid-liquid interface,
b is the effective thickness of the liquid film surrounding the particles,
c = concentration of the solute in the bulk of the solution at time t,
$c_s$ = concentration of the saturated solution in contact with the particles, M is the
mass of solute transferred in time t, and $\dot{k}$ is the diffusion coefficient.
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Now, coming to the mass transfer in leaching operation so, we have mentioned that it is a molecular transfer process. So, the equation of mass transfer we can write in this way that dM by dt that is equal to constant k dash in to the interface area through which the transfer of the molecule is taking place into C s minus C by b, where C s is the concentration of the saturated solution in contact with the particles ok. And, C is the concentration of the solute in the bulk of the solution at time t.

So, so C s minus c that will work as a sa a driving force, A is the interface area and b is the effective thickness of the liquid film surrounding the particles. And, mass of the solute, M is the mass of the solute, M is the mass of the solute transferred in time t, and k is the k dash is the diffusion coefficient. So, this is how having a deficient means the molecular transfer process considering it as a molecular transfer process we can have this equation to find the rate of mass transfer. (Refer Slide Time: 31:05)

Mass transfer in leaching operation:	
$\checkmark$ For a batch process in which V , the total volume of solution, is assumed to rem	ain
constant, then: $\mathbf{d}\mathbf{M} = \mathbf{V} \mathbf{d}\mathbf{c}$	
$\frac{dc}{dt} = \frac{k'A(C_S - C)}{bV}$	
The time t taken for the concentration of the solution to rise from its initial value	ue c <sub>0</sub>
to a value c is found by integration, on the assumption that both <u>b</u> and <u>A</u> remai	n
constant. Rearranging: $\int_{c_0}^{c} \frac{dc}{c_s - c} = \int_{v_s} \frac{k'A}{Vb} dt$	
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For a batch process in which V, the total volume of solution is assumed to remain constant then, we can write this as the mass that that will be transferred that is equal to volume V the volume V of the solution into dc. So, that is the concentration gradient. So, concentration generally we are expressed in kg per meter cube like that and the meter cube so, ultimately we can get the change in the mass.

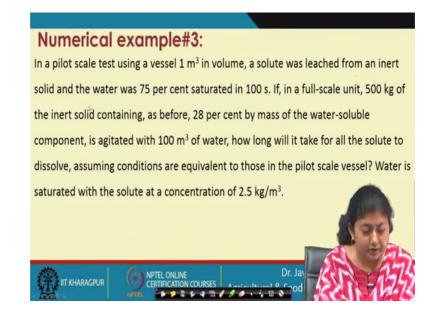
So, dc by dt that will be equal to k dash into A C s minus C by b into V. So, this is concentration gradient the time t taken for the concentration of the solution to rise from its initial value of c 0 to a value c is found by integration, on the assumption that both b and A remain constant. So, the thickness of the liquid fill will remain constant and the interface area, the contact area through which the transfer is taking place between the solid material and the liquid layer or liquid film; so, that will constant. So, from this we can write this equation integration c 0 to c dc by c s minus c that is equal to integration k dash A V into b dt.

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Mass transfer in lea	aching operation:
And,	$\ln \frac{c_s - c_0}{c_s - c} = \frac{k'A}{Vb}t$
✓If pure solvent is used initial	ly, c <sub>n</sub> =0
	$1 - \frac{c}{c_s} = e^{-\left(\frac{k'A}{bV}\right)t}$ $c = c_s(1 - e^{-\left(\frac{k'A}{bV}\right)t})$
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So, we are getting this equation as ln of c s minus c 0 by c s minus c. So, in pure solvent the concentration c 0 is initially taken as 0, there is no solute trace of the solute in that and this is equal to k dash into A V b into t. So, further from this we can write as 1 minus c by c s that is equal to e to the power minus k dash into A by V b into t ok. So, again c will be equal to c s into 1 minus e to the power minus k dash into A by V b into t. So, this is the equation of the change of concentration with respect to time in case of a leaching operation.

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So, let us see one example on that: in a pilot scale test using a vessel 1 meter cube in volume, a solute was leach from an inert solid and the water was 75 percent saturated in 100 second. If a full scale unit that is 500 kg of the inner solid containing as before 28 percent by mass of the water solvent component is agitated with 100 meter cube of water, how long will it take for all the solute to dissolve, assuming conditions are equivalent to those in the pilot scale vessel?

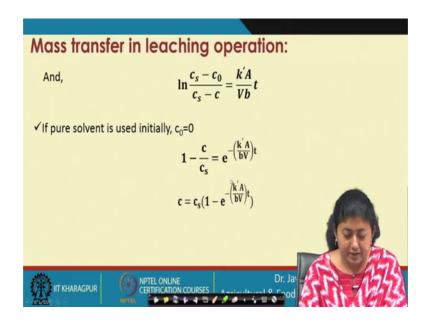
And, water is saturated with the solute at a concentration of 2.5 kg per meter cube. So, first we have using a pilot scale of 1 meter cube volume and the solute was reached from an inner solid and the water was 75 percent saturated in 100 second.

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ļ	Numerical example solution 3:
	Solution
	For the pilot-scale vessel:
	c = (2.5 × 75/100) = 1.875 kg/m <sup>3</sup> ; $c_s$ = 2.5 kg/m <sup>3</sup> , V = 1.0 m <sup>3</sup> and t = 10 s
	$1.875 = 2.5(1 - e^{-(kA/1.0b)100)})$ and: $kA/b = 0.139 \text{ m}^3/s$
	For the full-scale vessel:
	c = (500 × 28/100)/100 = 1.40 kg/m <sup>3;</sup> c <sub>s</sub> = 2.5 kg/m <sup>3</sup> , V = 100 m <sup>3</sup>
	Thus: 1.40 = 2.5(1 - e <sup>-0.139t/100</sup> )
	t = 591 s (9.9 min)
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Now, we will see that for the pilot scale vessel the concentration level will be 2.5 into 75 by 100 ok. So, we are getting 1.875 kg per meter cube, this is the concentration and c s in the saturated solution concentration is 2.5 kg per meter cube, volume of the pilot plant is 1 meter cube and time is 10 second right.

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So, putting this in the equation ok, in this equation c equal to c s 1 minus e to the power minus k dash into A by b into V because, some parameter is given in this problem and some are not. So, those are not given that terms will calculate from the pilot because, that ratio will remain same if you upscale this from the pilot to the actual scale. So, that ratio will make same and then we will calculate what will happen in case of actual system. So, will put it here in the equation value of c and c s so, 1.875 equal to 2.5 into 1 minus e to the power minus kA by V b so, V is 1 meter cube into b into time t.

So, kA by b we are getting this so, this ratio was required because, this is not given in the question and for the full scale vessel then concentration c will be 500 into 28 into 100 by 100. So, that the concentration is coming 1.40 kg per meter cube, c s is 2.5 kg per meter cube, V is 100 meter cube. So, if we try to get this what will be the time required? So, that will be 9.9 minute just we put the value of k by b in this equation ok. So, we will stop here and we will continue in the next class.

Thank you.