Aspen Plus Simulation Software – A Basic Course for Beginners
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Lecture-23
BTX Separation Through Distillation

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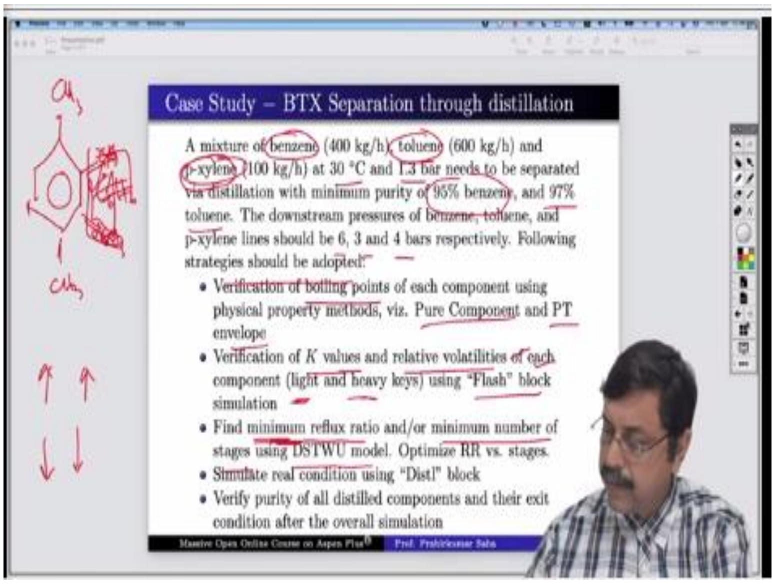

Case Study – BTX Separation through distillation

A mixture of benzene (400 kg/h), toluene (600 kg/h) and p-xylene (100 kg/h) at 30 °C and 1.3 bar needs to be separated via distillation with minimum purity of 95% benzene, and 97% toluene. The downstream pressures of benzene, toluene, and p-xylene lines should be 6, 3 and 4 bars respectively. Following strategies should be adopted:

- Verification of boiling points of each component using physical property methods, viz. Pure Component and PT envelope
- Verification of K values and relative volatilities of each component (light and heavy keys) using "Flash" block simulation
- Find minimum reflux ratio and/or minimum number of stages using DSTWU model. Optimize RR vs. stages.
- Simulate real condition using "Distl" block
- Verify purity of all distilled components and their exit condition after the overall simulation

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In today's lecture we will perform a case study on BTX separation through distillation. BTX means benzene, toluene and xylene. Benzene, as you know its structure is something like this C_6H_6 and whenever we have a CH_3 group, a methyl group attached to it, then it becomes toluene. And when there are 2 methyl groups either in the ortho position or in the meta position or in the para position, it is called xylene. So, this is ortho-xylene, meta xylene, and para-xylene.

In today's lecture we will work with para xylene, so we have a structure of this nature. The mixture of benzene, toluene, and xylene with flow rates are given the temperature and pressure is also given, so this mixture needs to be separated through distillation. These are very common aromatics and benzene, toluene, and xylene separation are very common phenomena in chemical and petrochemical industries.

But as you understand as a chemical engineer, it is not possible to separate 2 components from a solution if their relative volatilities are not favourable. In other words, if the boiling points of those 2 components are not far apart, one can't separate those two components through distillation. So, before trying to distill them using any unit operation in Aspen simulation, we have to do a certain property analysis of benzene, toluene and xylene and check their boiling points at the prevailing pressure that is 1.3 bar in this case.

If the boiling point of benzene, toluene, and xylene at 1.3 bar are far apart, we can surely separate them via distillation. And obviously, there should not be any azeotrope; azeotrope means when at a particular pressure or any particular condition, if the boiling points of both the components become same then it is called an azeotropic mixture and one can't separate them using distillation because their boiling points are same.

So, we have to be sure that they do not form any azeotropic solution and then only we can go forward and for all these analysis we have to take the help of Aspen property analysis. Mainly we will do the pure component analysis and also we will do PT envelope. This benzene, toluene, and xylene need to be separated via distillation with minimum purity of 95% of benzene and 97% of toluene, so we should ensure this purity level.

And a downstream pressure of benzene, toluene and xylene should be 6, 3 and 4 bars, respectively. That means the units where these separated components will be fed, need to be supplied at this pressure, so we have to ensure in our simulation. So, as we said that the following strategies should be adopted, first we have to verify the boiling points as I have described a few minutes back and then we have to verify the K values and relative volatilities.

And this information will give us the light and heavy keys among these benzene, toluene and xylene and that we can do with “Flash” block of Aspen simulation. And then we should learn about this minimum reflux ratio and minimum number of stages using DSTWU model. As you know in distillation, if we increase the reflux ratio and the number of stages then the quality of distillate becomes better but then number of stages increase and if you decrease both of them obviously the quality will go downwards. And there is some minimum value of both reflux ratio and the number of stages below which the separation is not possible. So, we have to find the minimum reflux ratio and minimum number of stages.

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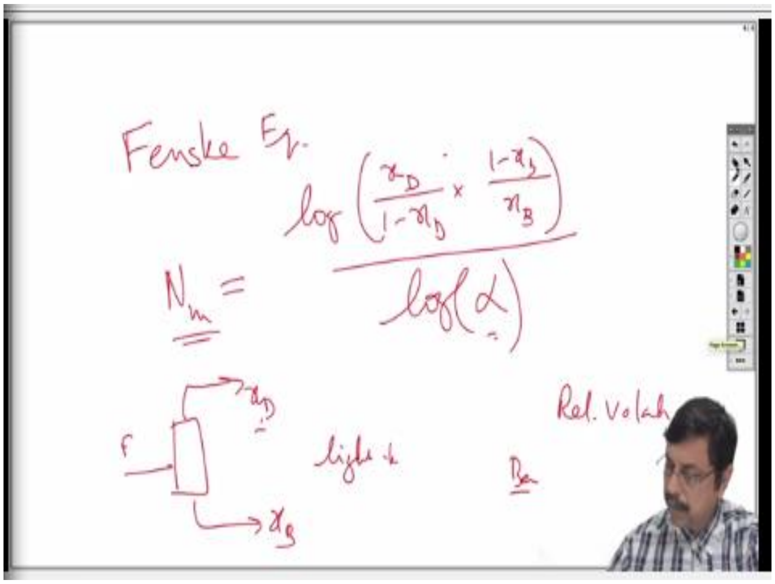

And we can use a Fenske equation, this Fenske equation So, this is the Fenske equation we can use to find out the minimum number of stages.

$$N_m = \frac{\log(\frac{x_D}{1 - x_D} \times \frac{1 - x_B}{x_B})}{log\alpha}$$

What is x D and x B? There is the distillate quality and the bottom product quality, where x is the mole fraction of the l key. So, if benzene is the light key then x_D is the mole fraction of benzene in the distillate and x B is the mole fraction of benzene in bottom product, this is the feed and what is alpha? Alpha is the relative volatility and all this information we will get from “Flash” model.

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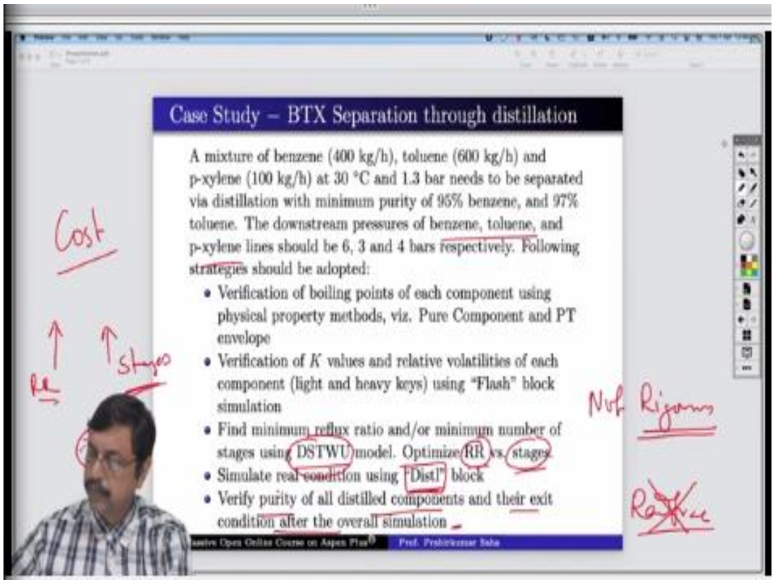

Case Study – BTX Separation through distillation

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Then we have to optimize this reflux ratio and stages because as we said that if you increase the reflux ratio and the number of stages then your product quality will be better. If you increase one of them and decrease the other then the product quality will remain same, we can control the product quality by changing them. So, if you increase them, you will obviously increase the cost of distillation, so we have to come with a trade-off between them.

So, we have to plan something in such a way that the cost is within our limit, so that we do not have to use too many stages and reduce our cost. And already we have learnt about DSTWU model a few lectures back and you know that this is a not so rigorous model. But if you need a rigorous calculation you have to use "Distl" block or more rigorous fractionation can be done with RADFRAC model.

But in this particular case study we will not use RADFRAC model, we will use only "Distl" block. So, we will initially do some rough calculation with the DSTWU model to find out the number of stages and the reflux ratio. And then we will do the simulation at real conditions using "Distl" block because this is more rigorous. And at the end we have to verify the purity of all distilled component and their exit condition after the overall simulation.

Because we have to know whether those downstream pressures are maintained whether the quality or the purity of the products are maintained, all these things we have to check before we end our simulation.

So, let us go to the Aspen simulation. **(Video Starts: 10:35)** So, this is the Aspen simulation window. First we have to enter the components, so let us enter the components benzene, toluene and p-xylene, they are very common components. So, if you add the names it will just identify itself. So, press next, now method we will use NRTL RK method. Because this is the best method with benzene, toluene and xylene, if you have any doubt you can just go through the literature we will find the same thing, press next, run it.

So, the properties are now run. Now we have to analyse the property analysis of benzene, toluene, and xylene. For that first, let us use the pure analysis, so the pure analysis is like this, now here we will adopt a strategy to find the boiling point.

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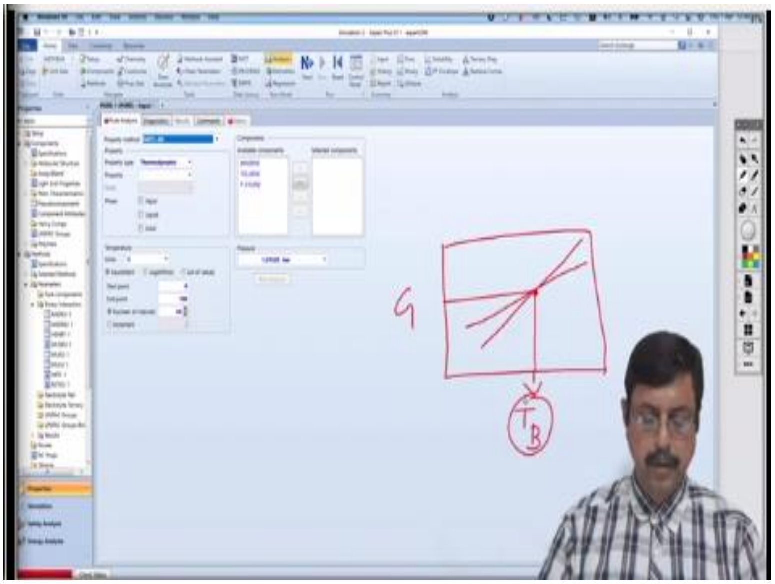

As we know that the best way of finding the boiling point is to find the Gibbs energy of both vapour and liquid lines. So, in point of intersection where the Gibbs energy of both vapour and liquid will be same, that is the point where we can say this is the boiling point of that particular pure component. So, individually we can try it out with benzene, toluene and xylene to find out the boiling points of those components. So, let us choose benzene and property Gibbs energy, this one is the gives energy of pure component will choose both vapour and liquid and let us use 1.3 bar.

Because that is the point where we have to do the distillation, so we will analyze everything at 1.3 bar. So, run analysis, so we find this is the point somewhere here. Now we can always zoom and check the value, so it is somewhere between, say 86 to 92, let us do the simulation within these 2 points. So, 86 to 92, run the analysis once again, yes, so it is around 88.4, so press right click and show tracker. It will show you the tracker and you can just pinpoint at the point of intersection and it will give you the value 88.4407.

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So, let us write it here 88.44 ℃ benzene at 1.3 bar. And we will record this information in this excel file because we may need it in future. So, it is 1.3 bar, for benzene, it is 88.44 ℃. Similarly let us check for toluene, it is obviously more than 100 ℃, let us check 120, run analysis. Well, so let us say from 117 to 130, run it once again. Yes, so again you just press show tracker and the exact value will be somewhere over here, so it is 199.64.

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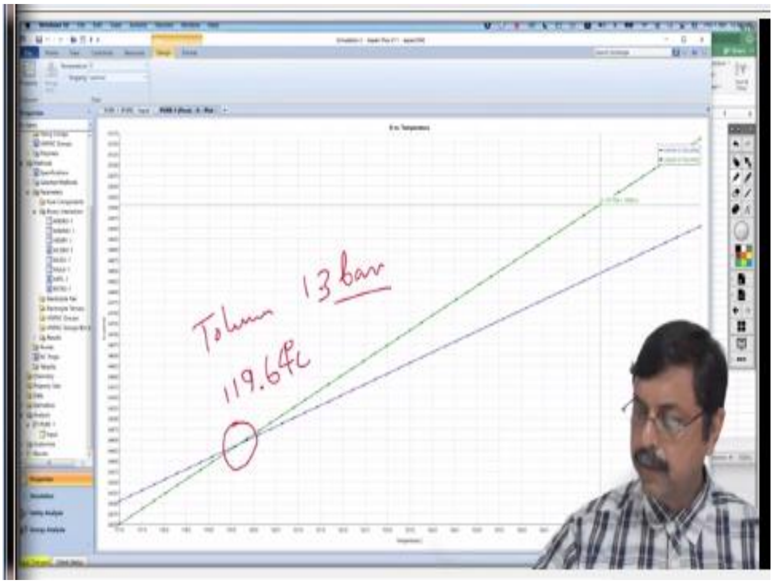

So, this is the point it is 119.64 °C toluene at 1.3 bar. So, let us write this information once again, for toluene we have 119.64, for xylene let us add it to 160 and let us zoom it between 147 to 149. So, it is 147 to 149 maybe it is somewhere at 148, so with the tracker you can pinpoint it at 147.94.

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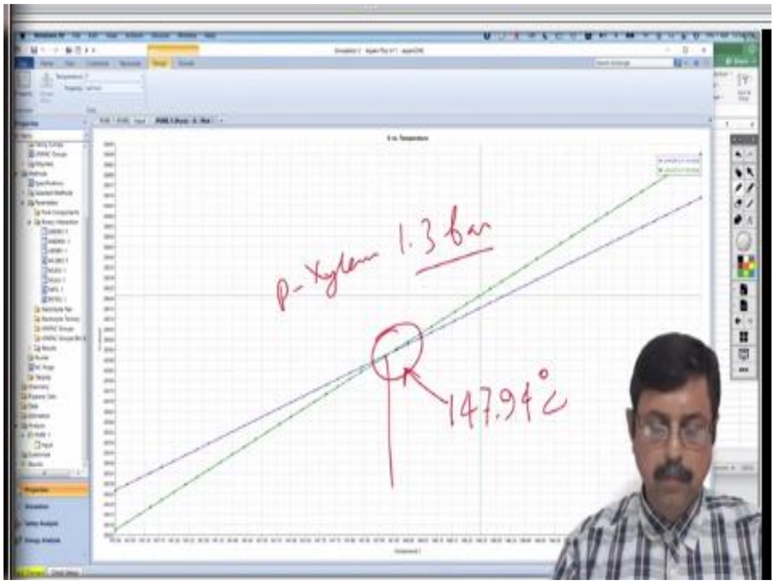

So, it is 147.94 °C, this is for para-xylene 1.3 bar, so we have this information let us again write it 147.94. It clearly shows that the boiling points of benzene, toluene, and xylene are far apart at 1.3 bar pressure. So, it is possible for us to distil them at that temperature. But we never know if the pressure changes there might be a possibility of forming azeotrope. So, let us go and check it with PT envelope because PT envelope is a more comprehensive way of analyzing the VLE.

For that we will go to the PT envelope curve, let us hold this pure envelope analysis, let us deactivate this one for the time being. And PT envelope input, let us first do it with benzene only as a pure component and run the analysis. And then let us bring once again PT envelope 2, run for toluene, this is for toluene and bring the third PT envelope, which is for xylene, run analysis. So, we got 3 PT envelope, all for benzene, toluene and xylene, we have got V frac 1. That means the boiling point curve has been obtained. So, this one changes the name to benzene, changes the name to toluene, and the third one lets it be p-xylene.

So, merge this plot with first let us take this one, merge it with benzene plot, go to plot number 3 again merge it with benzene plot. So, these plots have all 3 curves and let us make it a single axis, so this is the single axis y, yes. So, we get the boiling point curve for benzene, toluene and xylene from 1 bar pressure to 34 bar pressure and nowhere they cross each other. So, we have distinct boiling points for each and every pressure.

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For instance we are operating at 6 bar then the boiling point of benzene will be around 150, boiling point of toluene will be around 190 and the boiling point of para-xylene will be around 220. So, they are again far apart at 6 bar pressure. So, they do not form any kind of azeotrope and we do not have any problem with distillation. So, with this idea let us go forward. So, the first job is over, verifying each component's boiling points using physical property method.

Then we have to verify with K values and relative volatilities of each component the light and heavy keys using “Flash” block simulation. For that we have to go to the simulation environment So, property analysis is done, now we go to simulation and we will take a “Flash” tank, this is the material in, this is vapour out, this is the liquid out. Rename them, this is feed, this is vapour and this is liquid, press next.

We have to give the information about the feed; it is at 30 ℃ 1.3 bar, so it is 30 ℃ 1.3 bar pressure. And the flow rates benzene 400 kg per hour toluene 600 kg and xylene 100. So, 400, 600 and 100 at kg per hour, so it is mass flow rate, so this is mass flow rate kg per hour 400, 600 and 100, press next. So, we need to run the “Flash” pressure is 1.3 bar but the temperature. What is the temperature?

We have to run them at temperatures which are actually the boiling point of benzene, toluene and xylene and check the K values. So, let us run at 88.44 ℃ for benzene. So, write 88.44 ℃ and run the simulation, go to the results and see the phase equilibrium. So, this is the phase equilibrium information that we have with us, what we will do? We will snip this and then we will keep it over here, so this is about benzene.

The same thing we will do for other 2. So, now let us run it for this next it is 119.64, so let us write at 119.64 ℃, run it, go to the results, and check the phase equilibrium. Again let us snip, we will need this information later. So, this is for toluene and finally we will do the same thing for xylene. So, the temperature is 147.94, so 147.94 and run the simulation. Once again go to results, check the phase equilibrium and here again we snip it and keep it over here, this is for p-xylene.

Now do not get confused between them because we are not exactly doing it for any component, we are doing it for the temperature at which those components have a boiling point at. So, we are doing it at 88.4 ℃ because this is the boiling point of benzene. So, we have done all this calculation at 119.64 because this is the boiling point of toluene and so on. Now we have all this information with us, now we will have to do some kind of chemical engineering calculation rather than simulation.

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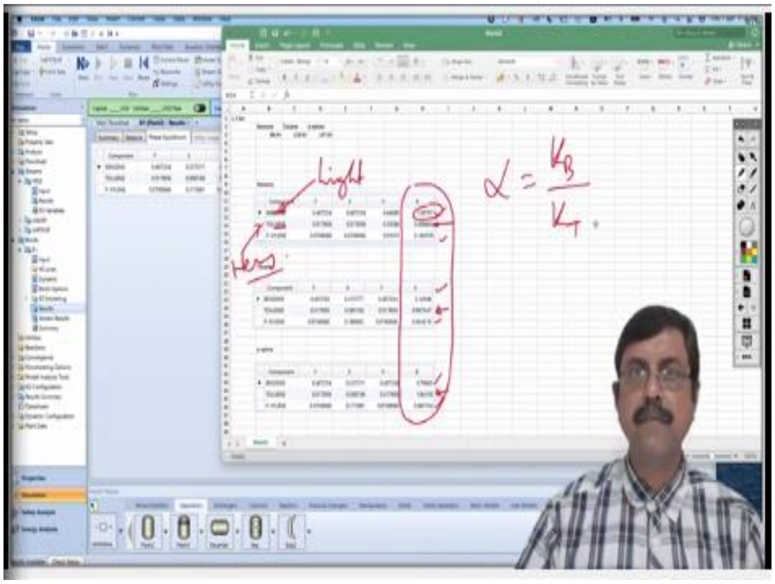

Now here are the K values that we have got and we can understand that for every simulation, we have the highest K value is for benzene, lowest K value is for xylene and for all of them toluene is the middle one. So, this is consistent for all the temperatures, so even if you do not do any property analysis from here, you can also understand that they are not forming any kind of azeotrope in this temperature range.

From that, you can understand that the K value of benzene is highest means this benzene has to be the light key and toluene is a choice for heavy key. So, benzene becomes the light key and toluene becomes the heavy key, xylene we will take care of later. Now what is the relative volatility? Relative volatility, in this case will be K value of benzene by K value of toluene that means the K value of light component by the K value of heavy component.

So, this calculation we need to do, so we write it over here, so we have 1.00707 and here 0. 408843, this one is 2.16368 this one is 0.997547 and the last one is 3.79945 and 1.94139. So, these are the K values of benzene and toluene respectively for 3 temperatures. So, this one, what is the alpha value? The relative volatility will be this by this, so this by this, so it is 2.46 in the first case for 88.44 °C is like this.

For other cases just stretch and you will find 2.16 and 1.95. So, if we take an average of them, we will find 2.19 or if we want to take the geometric average, we will see it is this into this into this to the power 1 by 3 because there are 3 components. So, both arithmetic and geometric average they are almost same. So, we have to choose the value, let us choose the value 2.19, so we take the average relative volatility as 2.19. So, this is our alpha value, that is relative volatility, we can say relative volatility. So, we can write relative volatility is 2.19, so this is one we will use. Now next comes the x D and x B.

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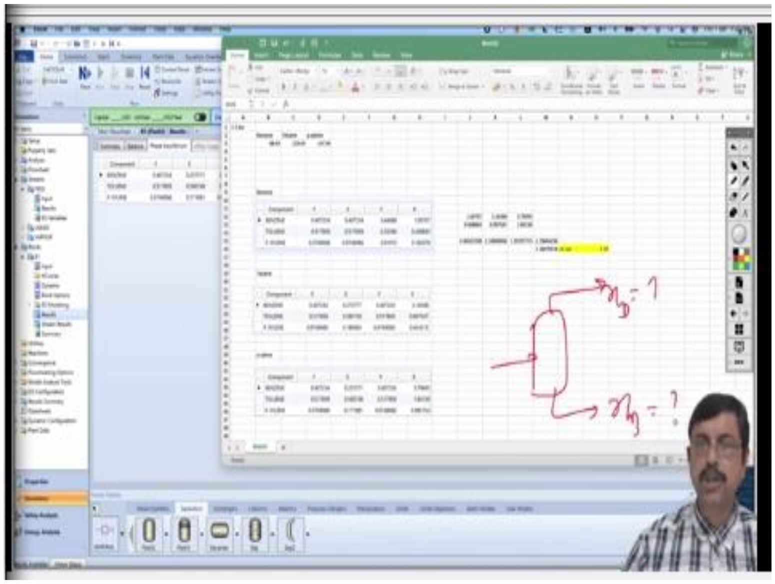

That means what is the desired value of benzene at the top and at the bottom? We need a minimum purity of 95% of benzene at the top. So, we can say that x_D will be 0.95, if you have the value of x_D, x_B, you can always calculate from mass balance or component balance. We do not have the figure right now, let us avoid the calculation at this moment, let us take a value wild guess. It does not matter what you take, you just take a low value because, anyway Aspen plus will do all those calculations.

You must wonder why I am doing this because I want to use the Fenske equation to calculate a minimum number of stages, so I am doing all these things for that. So, for that we know the minimum number of stages,

$$N_m = \log(\frac{x_D}{1 - x_D} \times \frac{1 - x_B}{x_B})$$

that is the numerator of Fenske equation and denominator is again log of the relative volatility. So, we get 6.96, so 6.96 is the minimum number of stages required for the distillation to take place.

Now let us use this information in our following calculation. For that let us go back to Aspen and let us delete this. So, we have deleted that block instead of bringing in the DSTWU block, so as you might remember, DSTWU is a shortcut distillation method block that uses Fenske Underwood Gilliland method. So, you just connect the feed stream over here, connect this thing over here and connect this thing over here.

Now it is a total condenser, so it is no longer vapour we should not use a misnomer, so it is liquid only. Let us call it benzene because it will be purely benzene only, so let us say benzene and this is a mixture of toluene and xylene so let us write tol-xyl, we will separate them later, press next. So, this is the information it is asking, now let us use 6.96, we know that the minimum number is 6.96, let us use 7 stages.

So, either you can use reflux ratio or minimum number of stages. As we have calculated the minimum number of stages using Fenske equations, so we are giving this information. Aspen itself will calculate reflux ratio. The condenser and reboiler our process is operating at 1.3 bar, so the condenser will be little less than that and the reboiler will be little higher than that, so let us take it is 1.2 bar and it is 1.4 bar.

The light key component already we have checked is benzene, and the heavy key component is toluene. The recovery light key recovery will be 0.95 set and toluene heavy key the recovery we said it is 0.075, so let us write 0.075 and run it. Now it did not take much time, so directly go to results. Here you see the minimum number of stages they have calculated 6.81295 and our calculation shows 6.96, they have calculated 6.81.

So, it is close enough, number of actual stages 7, minimum reflex ratio 1 and actual reflex ratio is 59, now this figure is not at all good, we are not happy with this figure. So, let us redo the calculation with some other values, maybe 7.5% recovery might not be the correct value, it may be much lower than that, so let us try 0.03 instead and check what is the figure. Go to the results, now the reflux ratio has come down quite significantly.

So, you can understand by giving proper information we can significantly reduce the value of reflux ratio and that can be within our acceptable range. And with that bargain minimum number of stages has increased to 7.93 and actual number of stages they have set 15.8795. Now let us go and see the stream results. So, this is the stream result, see the mole fraction of benzene, yes, so it has come to 96% of benzene over here. And in this line it is 0.03 that means at the bottom that mole fraction of benzene is 0.034.

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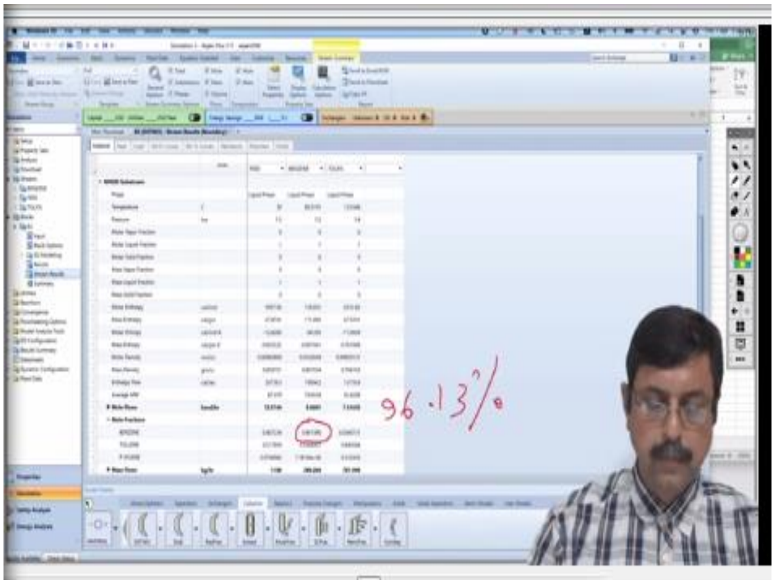

So, we have arrived at 96.13% purity which is very much within our limit. Because we wanted minimum purity of 95% benzene and we have got 96% of benzene, so we are happy with this result. So, simply what we will do? We will go back to these results and then we will snip this information because this information will be required for us. So, we have this information with us, what we will do?

We shall again knock off this DSTWU, and instead we will bring in the distl because that is more rigorous. So, we have optimized reflux ratio and number of stages we will simulate with the real condition using “Distl” block. So, for that again we attach them and connect the last one, yes, now run. So, it is asking for the number of stages, so let us see what information we have? Number of actual stages is 15.8, so let us use 16, so this is 16 and feed stage is at 9.16, let us use 9 only.

Reflux ratio, it is 1.686 distillate to feed mole ratio this is 0.402411, let us write 0.402, condenser pressure unchanged it is 1.2 and 1.4 respectively condenser and reboiler then run. So, go to the stream results, so here we find the actual value with mole fraction, it will slightly change well, now it has changed to 0.948, it has decreased. Because the shortcut distillation method was a rough calculation and this is the exact calculation and more rigorous, so it is giving 0.948.

And we are not happy with this because it is not solving our purpose; it is going below 95%. So, we have to do certain calculations, we will say increase the reflux ratio to 1.7 or maybe 1.8 say and let us check the stream result once again. And we find the mole fraction it is 0.95, yeah, now we are happy that we are getting the value over there. But what is the outlet pressure? The outlet pressure is 1.2 bar, but the stream says that the downstream pressure of benzene has to be 6 bar.

So, we have to use a pressure changer but here the vapour fraction is 0 that means completely liquid. So, we should use pump, if this line contains vapour, we have to use compressor but right now as it is liquid fully liquid, so we have to use pump. So, use the pump over here, connect this stream with the pump, and the pump outlet is here. So, right here as bench and press next, it will ask for the pump information.

So, discharge pressure is 6 bar, you can use pressure increase, pressure ratio also, you can enter the information about pump efficiency. For the time being let us not go into the details, just run. And now you can see the final value of benz, if you see the stream summary, so benze here the pressure you can see from here itself, pressure is 6 bar as expected.

And mole fraction we have not changed anything, so it should remain at 0.95, so we are happy with the benzene flow. Now our job is not over, we have to separate toluene and xylene, now here also we have to follow the same procedure. That is here again you have to do the “Flash” calculation for that I have to delete everything and start afresh to perform the “Flash” calculation and then again you have to come back to this.

So, I leave this exercise for you to practice, what I will do right now? I will start with an assumption and let us check whether the assumption will take us to the desired value of product quality of toluene and xylene. So, let us begin from the DSTWU calculation. So, let us connect it over here, this is the feed and these are the material outlet and let us rename them, let us say this is our toluene and this is p-xylene, press next.

So, it is asking for the number of stages, we do not know actually let us begin with say 20 stages. The condenser pressure and reboiler pressure as you can understand that the information is given the toluene and xylene lines downstream pressure should be 3 and 4 bars respectively. So, that actually means that here it should be 3 bar and here it should be 4 bar. So, we will operate this at 3.5 bar, for that we have to insert a pump over here.

So, let us take the pump over here, we will take this reconnect destination at this pump and pump outlet will connect with this column. And we will press next, the number of stages it should be 3 bar, it should be 4 bar, the light component obviously it will be toluene, the recovery of toluene should be somewhere at 97%. So, we write here 0.97, heavy key is xylene and we do not know the recovery let us write 0.01, let us see what happens?

Next, so we have added one pump, the discharge pressure we will say it is 3.5 bar. Because if it is not 3.5 bar we will not be able to operate it at 3 bar and 4 bar here, so discharge pressure is 3.5 bar. So, the feed will enter this column at 3.5 bar, press next, ready to run the simulation, yeah, so no error. Now let us see the result, now we have not renamed them, let us rename them, so that we can understand what we are doing?

Let us write it as Distil, no it is DSTWU, this one is distil, so we write here distl 1. So, this is the benzene column and this is the benzene pump, this one is the. Here it is toluene-xylene pump, so we keep these names over here and here. So, just run once again, now these are the blocks for results we check the results of toluene and xylene, DSTWU, these results.

Here the reflux ratio is 2.31, minimum number of stage 12 and actual number of stage 20 we have given but what is the stream result? The stream result is that the mole fraction of toluene line is 0.95 but we ask for 0.97. So, now please look at this figure, molar flows and mole fraction. Now here you can see the benzene content in the S 7, S 7 means we may rename it actually, let us say the TPLPOUT, let us give this name.

So, the toluene pump out, this is the feed to the column that separates toluene and xylene. Now that particular feed is already having 0.303 kilo mole per hour benzene which has not been distilled in the previous column. So, this benzene is coming into the toluene-xylene stream and this toluene-xylene stream is containing this benzene, this benzene is having a mole fraction of 0.40, that means 4 mole% of benzene is present in the feed stream.

Now if the same benzene is distilled again in the second column, obviously, if the toluene has to go higher as the light key in the second column, benzene will also be distilled along with it because benzene has lower boiling point. So, the toluene line that means the distillate line in this column it will contain toluene + benzene among which benzene already has this much of kilo moles per hour whose mole fraction is 0.047.

That means 4.74% of benzene is always present there in the toluene line, the distillate line. So, if 4.7 mole percent benzene is already present in the distillate line then in the same distillate line the percentage purity of toluene can never be 100 - 4.74, that means 95.26%. So, that means we can never go beyond 95.26% of toluene purity in the second column in this kind of situation. But in our problem, we are asked to get a minimum purity of 97% toluene, which is impossible.

In this present setup it is not possible until and unless we chuck out some more benzene out of this toluene-xylene stream and send it through the first column. I mean we have to increase until unless we increase the efficiency of the first column and drag out more benzene out of it, we cannot reach 97% toluene purity in the second line. No matter how many stages you employ or to whatever extent the reflux ratio you increase it is never possible to arrive at 97% toluene.

So, in such cases for instance if you are content with 95.1% purity of toluene then you can just check the value of pressure over here, it is 3 bar pressure and it is 4 bar pressure in the line which exactly we are expecting 3 bar and 4 bar respectively in the downstream pressure. So, you can just simply change this block with a “Distl” block but before that we have to remember the values of the results which are exactly actual reflux ratio, this information will be needed.

So, we can just snip this value, so just keep it over here and then we can simply delete this and instead we insert a distil column over here, connect these streams, connect this stream and then connect this stream. And we have to rename this as Distl 2 and we have to give this information number of stages. So, we already have snipped let us go and find the snip, so we have number of stages 30 and feed stage 16.93, so we can take 17, so 30 and 17.

Then reflux ratio, it is 1.46, distillate to feed mole ratio, it is 0.8509, so just write 0.85, condenser pressure 3 bar and reboiler pressure 4 bar, run it. And go and see the stream results, toluene it is mole fraction is 95.12 as expected. And if you see the outlet pressure 3 bar, this is 4 bar and 6 bar. **(Video Ends: 1:05:50)** That matches with our expectation of downstream pressures of benzene, toluene and para-xylene lines. So, we have successfully verified the purity of all distilled component and their exit condition after the overall simulation.

Obviously we cannot reach 97% toluene that is just a mathematical impossibility, we have explained it properly. So, by this manner you can use the physical property method for analyzing whether the separation through distillation is possible. And if it is possible then how it is done? How you can use “Flash” tank for analyzing the relative volatility? And then you can use DSTWU for shortcut distillation and “Distl” block for more rigorous calculation you can do. So, with that we end our lecture at this point, we will continue with another case study later. Thank you.