

Principles and Practices of Process Equipment and Plant Design
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Module - 02
Lecture - 16
Practical issues in designing distillation processes

Good day to you all. We are going to talk about certain Practical Issues in designing distillation processes. So far, what we have covered are the basics of engineering design and process design. We have followed with a special type of separation process design which is designing of distillation separation, where we have talked about flash distillation or flashing. This has been followed with fractionated and fractionated designs.

At the last class, you have been given input on the basics of batch distillation and its design and operation to some extent. Today, what we are going to decide is about the practical issues normally that is faced by the designer while he is engaged in designing such distillation separation processes to start with.

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Practical issues often faced by the designer

- Sequencing multicomponent separation
- Process selection: Flash/Fractionation/Batch
- Optimum design configuration
- Functional aspects of the Design Output
 - Erection, Commissioning
 - Operation: production phase, start-up, shutdown
 - Flexibility aspect of the finalised design – stable design

$A, B, C \rightarrow A, B, C$

The slide features a background with various engineering icons like gears, a tree, and a circuit. In the bottom right corner, there is a small video inset of Prof. S. Ray. The NPTEL logo is visible in the bottom left corner.

He is supposed to handle multi-component separations. They have a mixture of A, B and C. This is in the order of decreasing volatility and perhaps he is supposed to produce three

streams rich in A, rich in B and rich in C. So, the question comes that what should be the sequence of separation and what is the option that we have to cover in this course because this is a very common situation which is faced by the designer.

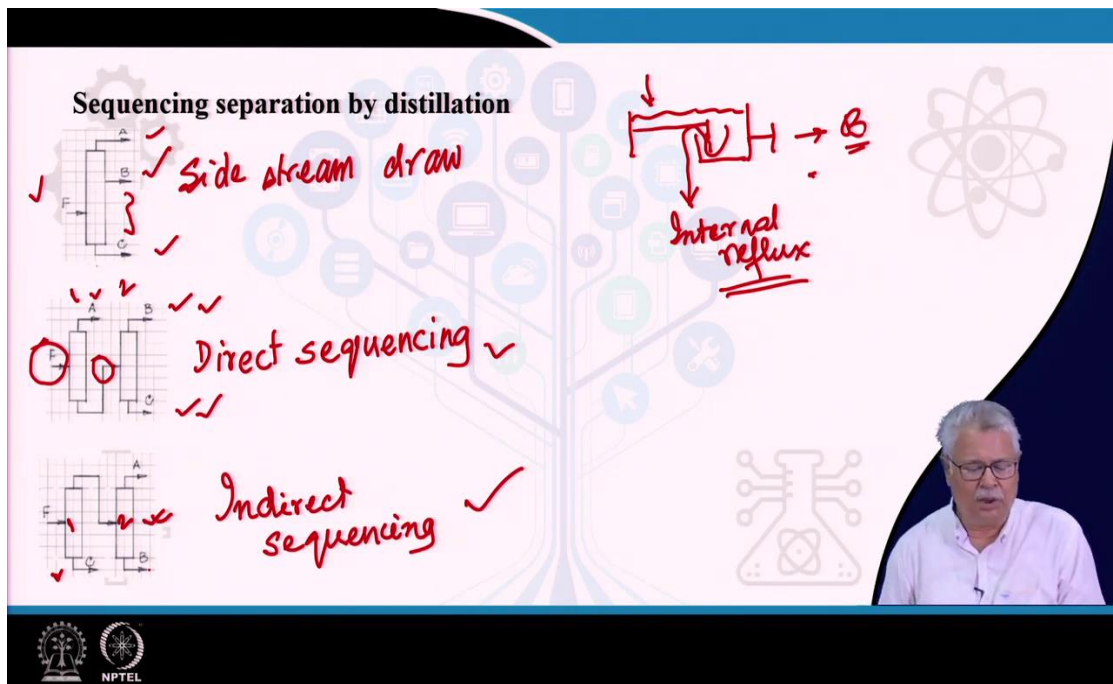
The next option that comes is almost equally important that selecting the process; when to select flash, when to select fractionation, when to select the batch distillation. The inputs on these have already been given to you why these topics were discussed. But, I will try to consolidate here and include a few heuristics on that.

We have to talk about optimum design. That means, just delivering the functionality is the basic requirement. But it has to be delivered efficiently, particularly with an economic consideration which is the essence of any engineering design and differentiates the engineering design from the science-based design.

We certainly have to take care of the functional aspects of the design output. The plant that one designs the distillation setup that design has to be erected. It has to be commissioned safely, economically with the available resources. It has to be produced efficiently; that means, it has to be operated in the production phase and which means there will be startups and shutdowns. So, all these facilities have to be there in the process that you design.

So, your distillation process should consist of considerations for erection, commissioning, production phase, startup and shutdown. Typically the startup and shutdown required certain additional facilities which possibly will not be used during the operational phase. In the end, you definitely will be arriving at a finalized designed output and that you have to judge that how good your final output is; whether your design is stable or not that also we will be covering today and having an idea about it.

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To start with we start on today's first topic which is sequencing multi-component separation by distillation.

The first configuration that you see here is basically what? It uses a single column with a side stream draw. That means, the components rich in A is the top product, the component rich in C is a bottom product, and possibly there will be some composition on a tray that will meet the requirement of the stream as a product with an adequate concentration in B.

Let us look at the advantage of using a single column. Obviously, your involvement in finance is less; that means, you require a minimal amount of investment because you are going to use a single column. But, your operation is slightly more complicated, particularly controlling the quality of A, B and C together it requires a little bit of in-depth knowledge of the distillation process itself.

But, in any case, we already have dealt with simple columns which produce only a top and a bottom product and more or less a similar process can be used in case of this particular side stream draw columns. One thing you must remember is that this quality of B depends on how much you draw as a side stream.

From the side stream draw tray, if this is your side stream draw tray which I am drawing here like this and the side stream will be drawn here and whatever you do not draw will be falling here as internal reflux and this is your stream B.

You will notice one thing, whatever liquid falls on this if the entire amount is drawn out as B. The internal reflux would dry out. That means, it will make these trays ineffective; that means, there is a limit up to which if you have to maintain a quality curve B you can draw. That is slightly more complicated as compared to operating your straight column.

Often there are other options of separating A, B, C mixture into streams reach in A, B and C. The next one is here which is called direct sequencing. This definitely and it is very obvious that it is using two columns; the first column and the second column. The first column top product is the lightest which is A. The bottom from the first column goes to the second column which is again split into two streams, which is again a fractionator. And, it is B which is produced at the top of the second column and whatever remaining is your product C.

That means, here there is one big advantage perhaps you should ask me that why to use one additional column. The first advantage that you have is you can check and change the split between this stream (B) and this stream (C) more easily. Because this stream particularly becomes independent. If you want to have a larger variation in the quality of B, it is possible in this direct sequencing operation.

There are quite a few other advantages also. That is if you look at the throughput of column one the feed is the entire quantity F whereas, the second column has got a much smaller feed. That means you expect the second column to have a lower diameter or a thinner column which is expected to be cheaper.

In some cases where you want greater flexibility between the split of B and C products. You have a large amount of a presence there that makes the second column substantially smaller in diameter, in such cases, direct sequencing is definitely of help. So, we know now that the first option that comes the cheapest possibly is a side stream draw, the other option is the direct sequencing.

Similar to direct sequencing it is also possible to have indirect sequencing which is this particular option. In indirect sequencing what we take first is the bottom from the first

column which is this stream C. The top product is a mixture of A and B which is split in the second column.

The same logic applies here too. What you have if you take that large amount of C to get out from your mixture, you are left out with a very small quantity of which is a mixture of A and B primarily which can be split into two streams in a well-controlled fashion in your fractionator too.

So, one advantage of this is or rather by looking at the direct sequencing and indirect sequencing, we can see that such a sequencing method becomes very useful in reducing the investment cost when one of the products is in large quantity. So, if you take out the large quantity, first the later columns that you have to add are usually of a smaller diameter and that helps the economics.

But, in any case, this could be an advantage only when the separation is rather important between either B and C in the case of direct sequencing and A and B in the indirect sequencing case.

So, I would like to say that though I have given you an example of fractionation where the sequencing is implemented, a similar thing is true in the case of batch distillation also. In case of the batch, you can produce in a set of accumulators products which are rich in A, B and C or you can run a batch take out the product from the top and the bottom and rerun either top or the bottom, similar to the direct sequencing or indirect sequencing. Naturally, these are the options.

In the case of batch distillation, the side stream draw is not very economic. Because as such batch separation or batch distillation is used when the extent of separation required is not very high. We move forward to the next thing.

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A set of heuristics rules and information -

- Small capacity: usually batch distillation ✓
- Batch distillation: frequent product/feed product quality changes ; Better control ✓
- Batch distillation: best for separation of volatile product / impurities. }
- Flash distillation: a cheap alternative when purity is not a major concern } *Single*
- Economic optimum option needs to be choose among best optimised options of all alternatives } *FUG method*
- Shortcut design procedures are normally sufficient to evaluate/compare economic optimum }

Flash, Fracn, Batch

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Have a look at a set of heuristic rules and certain information. These heuristics will firstly, try to relate what to choose from batch, fractionation or flash. You already have given input while being taught in the batch distillation process that it is usually economic in a small capacity.

This requires startups and shutdowns because you have to fill your batch still, run it, shut it down and make it ready for the next batch. So, quite naturally if you lose by off-spec, you lose one batch material only as off-spec. So, your better control is always there in the case of batch distillation systems.

But at the same time, you can have the flexibility of changing the product and the feed quality depending on what is demanded and what quality of feed is available to you for the same set of equipment. Quite often batch distillation in presence of such uncertainties in the feed and the product quality specifications is quite important and efficient.

You will also look at another case, where batch distillation is important. Very often you will find some streams are contaminated with either a high boiling or a volatile product. Say a heavy oil is mixed with gasoline, the boiling point difference is substantial and the quantity of gasoline is small. So, in such cases possibly, you will simply use a batch distillation which you will be using either a single state batch still or maybe one with

multiple stages. But, it will be effective in separating when the volatilities are substantially different and they are quite effective when you have separated the contaminants or the impurities.

You will notice one thing, the moment we say impurity the quantity of impurity is small. So, usually, you will find batch distillation being used for systems where, one product dominates compared to the other, like either your distillate is small or what you are leaving at the end is also substantially small.

But, remember while distilling something in batch you have to ensure that your heating element has to remain submerged when the last drop comes out. That means, you cannot have 99 % of the distillate in a batch operation; possibly you can go up to some 15-20 %. That is a practical limit mainly posed by requiring the heating elements to remain submerged in the batch steel.

We also know that similar to the batch, the simplest form of distillation is a batch is a flash distillation. It is certainly the cheapest alternative, but we have a limitation here. That means the vapour and the liquid get separated using only a single stage. So, if your single-stage enrichment is sufficient for the feed, then you go for then only you can use the flash distillation.

But one big advantage is this is a cheap alternative, easy to operate. What you have to control is the flash drum pressure and we will see a little more details about this in the next few slides. The quality which is produced concerning the purity of the vapour it is may not be a very precisely controlled even.

We have a very important question in our hands. The economic option quite naturally the designer has to choose between options of flash, fractionation and batch. He has got a separation problem by distillation at hand. It is obvious that you have been taught that there are ways to optimize the fractionation, there are ways to optimize a batch and there are ways to optimize a flash also which we have not dealt with in detail.

But anyway, it is true and you have to appreciate it at this particular point. We do know that if I decide on a fractionator to be designed for a particular separation there will be an optimum and economic optimum design of the fractionator.

So, when you look at the best option, you should look at the best economic option of flash, the best economic option of fractionation and the best economic option of the batch and then only compare. So, basically the optimum or the final global economic optimum needs to be chosen among the best-optimized options; among the best-optimized options of flash, fractionation and batch.

Here is a big boon to us. While we are talking about multi-component fractionation, we have outlined a shortcut design procedure which is basically the Fenske Underwood Gilliland method. It is not only that you can have a separation of a multi-component distillation calculated by this method. It can also be applied roughly to binary systems. So, this particular method of design is quite sufficient to evaluate or compare the economic optimums.

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A set of heuristics rules etc. (cont'd.) –

- Practical (subjective) considerations often override computed economic optimum
 - ease of operation, proven / reliable design
 - similar plant in the same complex
 - ease of maintenance or common spare etc.
- Separation by distillation at lower pressures is easier
- A real life distillation column will always have some leakages through flanges, fittings, valve glands etc.
- Non-condensable (gases) are present in the system due to air in-leak, air dissolution in feed tack, cracking or other reactions
- Vacuum columns are preferred for high boiling point and heat sensitive components
- The temperature of the bottoms may be limiting for heat sensitive materials as bottom product

So, with this, we move forward in the list of heuristic rules and other things. The designer often will not opt for the most economic option. He has other considerations which may even override the economic optimum; the best economic option does not opt in certain cases.

In such cases, we are based on subjective considerations which may include ease of operation, proven technology and reliable design. Similar plants exist at the same complex. So, you do not have to train your manpower again on a new plant.

The ease of maintenance which you normally find whenever you are choosing a particular technology you contact its users, find out how is, how easy it is to maintain and run the plant, how good it performs in practice.

Definitely, if you have similar plants in the same complex you will have the advantage of common spare. For example, you need not have the same capacity column which is which you find to be optimum. You may go slightly away from the optimum design and have another column that is identical to the one which is remaining in your plant already so that you can use common spheres of heat exchangers, may be columns and other spare parts.

The thumb rule also tells us that the separation by distillation at a lower pressure is easier. It is true by considering the x-y diagram. If we look at the equilibrium curve in the x-y diagram, the equilibrium curve goes away further from the diagonal line as column pressure decreases. So, this is for decreasing pressure.

I am repeating this particular slide once more. We continue with the heuristic rules. Practical considerations often override the computed economic optima.

That means even if I have decided on a particular design of a plant we do not go for it. We may use certain other subjective considerations which could be ease of operation, which could be ease of operation, a proven or a reliable design to be adapted, similar plant in the same complex, ease of maintenance or the common spare etcetera. Separation by distillation at lower pressure is always easier.

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A set of heuristics rules etc. (cont'd.) –

- Practical (subjective) considerations often override computed economic optimum
 - ease of operation, proven / reliable design ✓
 - similar plant in the same complex ✓
 - ease of maintenance or common spare etc. ✓
- Separation by distillation at lower pressures is easier
- A real life distillation column will always have some leakages through flanges, fittings, valve glands etc. ✓
- Non-condensable (gases) are present in the system due to air in-leak, air dissolution in feed tank, cracking or other reactions ✓
- Vacuum columns are preferred for high boiling point and heat sensitive components
- The temperature of the bottoms may be limiting for heat sensitive materials as bottom product

What happens is at a lower pressure the x-y diagram deviates more from the diagonal line. So, if I have x here, if I have y here, in this direction this one will have a lower pressure as compared to this; that means, the pressure increase is this way. There is that means, this gives us one suggestion we should always try to design a plant which will operate at the lowest operating pressure, but there is a certain limitation to this which the designer considers.

In a real-life distillation column, there will always be some leakages through the flanges, fittings, valve glands etcetera. So, if it is an operating pressure which is above the atmosphere the content inside, the vapour inside will be leaking out and if it is operating under vacuum air will be leaking in.

So, the next point that we have to say is you will have the non-condensable gases are present in the system due to either air in-leak, air dissolution in the feed tank, cracking or other reactions. The vacuum columns are preferred for high boiling point and heat-sensitive compounds because it reduces the boiling point and the operating temperature of your column.

Naturally, the bottom temperature in a vacuum column is the highest. We definitely will be having a limiting maximum temperature for the column bottom temperature in case of

vacuum column and for other columns also. Particularly when the bottom product is a heat-sensitive material.

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Considerations during design –
(Safety; Economy; Resource availability)

Functionality

- Erection, Commissioning
- Operation: production phase / startup / shutdown
- Flexibility in operation
 - Variation in feed quality, product quality, design inputs
 - Design in presence of uncertainties in design input

The slide includes a handwritten process flow diagram in red ink. It shows a feed stream 'F' entering a system, passing through a pump, and then entering a distillation column. The column has a reboiler at the bottom and a condenser at the top. A reflux stream 'R' is shown returning from the condenser to the column. A distillate stream 'D' exits from the top. A bottom product stream exits from the bottom of the column. A handwritten note 'Start up line' with a checkmark is written near the feed line. The NPTEL logo is visible in the bottom left corner.

The basic considerations during the design for the designer are safety, economy and resource availability. The functionality that the design has to cater to are during erection, commissioning and the operation which includes the production phase startup and shutdown.

There is one more thing which is very important, the flexibility in the operation of the design that is finally, realized. It has to cater to variation and feed quality. The product quality demand and certain uncertainties in the design inputs. So, we have to be concerned about designing a plant in presence of uncertainties in the inputs to the design.

I just would like to add two things here. I will talk about the startup and shut down. In the case of a fractionator we normally have the column here. We have a condenser from where the liquid which condenses in the condenser comes to the overhead drum. From the drum, a part of it is taken out as distillate and a part of it is second sent in as reflux. It could be R or L whatever it is. I will also have here a reboiler, from where the vapour will be returned and we will be taking out a bottom from here and here is my feed.

Now, we have to have heating of my feed which means, the entire heating normally is not by the reboiler. The one reason is there is basically the highest temperature and this system is here. The feed is at an intermediate temperature. So, what you normally will be having is a heat recovery scheme that will go like this. It will recover heat from the bottoms and send the bottom out like this.

You have possibly other heating arrangements and exchanges in your feed line also. You definitely will be having a feed pump here which will be pumping the material which will be coming from some external stores possibly from a tank.

Now, when you start your pump and when you start your unit everything is cold. To have the temperature in your column what is done you usually will have a line drawn from here which will be drawn from here and connected to the suction of the feed pump. This is the start-up line. This is fitted with a valve. In fact, it is fitted with a globe valve and it is also fitted with a gate valve.

So what happens is, when you start your feed nothing vaporizes, so, it comes here. The entire thing is again drawn by the drawn from the bottom and is sent here. So, what you actually will be having here is one more pump which will be like this. So, this pump will be started once you have sufficient level here, it sends the entire thing back to the feed line which keeps on circulating.

So, what happens is with time since it circulates through the reboiler which has to be kept on by that time its temperature goes up, the vapour starts forming, it goes up the cold column condenses in the walls and the trays of the cold column and starts coming back. There will be after some time when the entire column will get heated and liquid will start coming into your reflux drum and your reflux drum will be having a level.

Quite naturally since some of the liquid which I had come here while it is circulating part of it gets transferred to the reflux drum you have to take a fresh amount of feed. During this period this startup line circulation goes on and gradually what you find; as you have the temperature in your top which corresponds to your distillate composition. What you do in that particular case is you gradually shut this particular, you gradually shut this particular flow and open the flow going to the storage of B.

So, this is how you start up fractionators. This is a very brief idea just to give you the fact that not only there are facilities for operating under normal conditions, but for your startup, you may require in your plant additional facilities like the startup line in your fractionators. I leave it to you to think that how will you gradually shut your plant down.

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Design in presence of uncertainties in design input

Flash Distillation system

Input

- Feed flow rate (F)
- Feed composition (x_f)
- Feed condition (T_f, P_f)
- Flash drum pressure (P_{flash})

Functional requirement (target): y_D

Output: Flash drum temperature (T_{flash}), Process scheme, P&ID, Drum size, Fabrication drawings

Measure of uncertainty : μ_i (mean) and σ_i (standard deviation) for input 'i'

Handwritten notes and diagrams on the slide include:

- A schematic of a flash distillation system showing feed F entering a drum, with bottom product x_B and overhead product y_D . A pressure P_{flash} is indicated.
- A graph showing a distribution curve for y_D .
- A handwritten note "Unique." with a checkmark.
- A handwritten note "92 108 10 F" with a checkmark.
- A handwritten note "|| $\frac{\Delta y_D}{\Delta F}, \frac{\Delta y_D}{z_f}, \dots$ " with a checkmark.

Possibly now it is time for us to talk about certain uncertainties in the design input. The flash design system will have design inputs as flash feed rate (F), x_f is a feed composition, the feed condition (T_f, P_f) and the flash drum pressure is also stated in this particular case. My functional requirement is after flashing my drum, overhead vapour should have a composition of y_D and, this is my x_B and this is my F .

Now, if you look at the design procedure the value of F , the value of x_f , the value of T_f , and P_f and the P_{flash} all are unique values. These are the best estimated unique values based on which you have given your design. That means, your x_B , your y_D that you have found out and the design dimension of your equivalent that you have found out depend on certain set of unique values of input that has been decided.

But, there will indeed be certain uncertainties associated with each of these. That means, if I have a variable here it could be feed, it could be $10 \text{ m}^3/\text{h}$, possibly this value this is 10.

But it could be 9.2 to maybe 10.8 and probably the maximum probable value has been 10 so this is 9.2 and this one is 10.8.

So, what I would like to say here is even though we use unique values for my design I have to test my output y_D after the design has been done that what is the effect of y_D , what is the effect of these inputs variations on y_D . That means we are supposed to report variations in y_D to the change in F , the change in y_D to changes in x_f and naturally so on.

As long as the changes are small it is easy and it is linear, and you can very easily either do a simulation or a repeat a hand calculation or we find out. But if you are finding that all these changes are not much effect, your y_D what you expect to get is a y_D which will have a distribution something like this. This basically is your y_D , this is a frequency or rather the probability of variation of y_D .

Quite naturally if you have a probability distribution of y_D because of the variations of these inputs like this or if you have another where it is much steeper this simply shows that this is a much more stable design. That means the design output is not affected by the input changes.

I think with this, I will be stopping and before that, I only say that the mean and the standard deviation for y_D is the parameter that is normally checked for this.

Thank you.