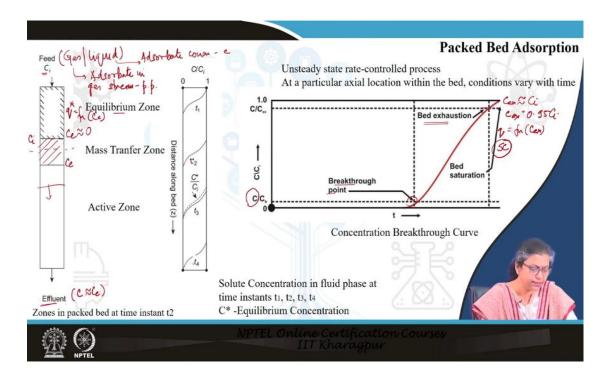
## Principles and Practices of Process Equipment and Plant Design Prof. Gargi Das Department of Chemical Engineering Indian Institute of Technology, Kharagpur

## Module - 02 Lecture - 32 Packed bed adsorption

So, hello everybody, once more we have gathered today to continue our discussions on Packed Bed Adsorption. I have already mentioned to you that the packed bed adsorption is the more conventionally used adsorber bed or rather an adsorber equipment or device which is used. As we have already discussed and it is quite evident to you that the packed bed adsorption is an unsteady-state rate controlled process.

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As I had started my discussions, we know that this packed bed adsorption can be used for different types of feed. The feed can be gaseous, the feed can be a liquid feed. Generally, if we have a gaseous feed, the concentration of the adsorbate in the gas stream is denoted by the partial pressure conventionally. For the case of a liquid feed, the adsorbate concentration is denoted by C.

Now, let us see the separation. I will just repeat what I had mentioned in the last class as a small recap and then we proceed further. What did we find? We find that in this particular case it is an unsteady state process. What does it mean? If you look at one particular axial location, we find that the conditions do not remain constant with time.

Why does it happen? At the start of the process what happens? We find that at the start of the process, the feed enters the bed. As the feed enters the bed, the mass transfer starts occurring. Initially, the mass transfer occurs near the inlet and the concentration of the adsorbent in the fluid phase decreases from the initial value of  $C_i$  to a value of saying  $C_e$  or whatever it is too much lower values which is almost equal to 0 a very low value. This occurs over a very narrow zone.

Now, initially, we find that this particular narrow zone is known as the mass transfer zone. The zone of the bed or the portion of the bed where the mass transfer occurs is known as the mass transfer zone. With the progress of time, what happens? Mass transfer keeps on occurring in that initial portion of the bed.

It keeps on occurring till over the entire bed we find the bed it becomes saturated and the adsorbate loading in the bed that becomes or that is equal becomes equal to q\* which is in equilibrium with the equilibrium concentration. The moment that happens, then that portion of the bed is no longer available for mass transfer, equilibrium has reached there, and that portion is known as an equilibrium zone.

So, under this condition, what happens? When the initial part of the mass transfer zone becomes saturated. It is not able to adsorb any further solute. It becomes the equilibrium zone. Then the feed stream with the unabsorbed adsorbate starts travelling further. As it starts travelling further, the mass transfer zone also starts moving from the initial part of the bed to further downstream.

Then the mass transfer zone shifts downstream to the equilibrium zone and mass transfer starts occurring here. So, therefore, after some time in this region, the initial concentration of the adsorbate in the stream is  $C_i$ , and here it is equal to  $C_e$ . The mass transfer continues to occur. After some time, this portion also gets saturated. Then when this happens, the mass transfer zone starts travelling to the lower portion.

Now, during this entire time, you remember that the mass transfer is occurring within the bed, and the effluent which is moving out that is almost devoid or that does not contain any very small amount or it does not contain almost any amount of the adsorbate.

So, therefore, the adsorbate free effluent whether the mass transfer zone is here, whether it is here, whether it is here, from the effluent which comes out we find that for this particular case your C is almost it is equal to C<sub>e</sub> which is almost negligible.

So, with the progress of time, we find that the empty set, it moves further. As it moves further, it leaves an equilibrium zone saturated with the solute behind it. Before the mass transfer zone, there will be some portion of the bed where mass transfer has not started at all. Now, this particular portion is known as the active zone.

So, gradually we find that the mass transfer zone. It starts at the beginning of the adsorber bed. It starts travelling downwards leaving behind an equilibrium zone. Before the upstream of the mass transfer zone, there is an active zone where mass transfer has not occurred. The mass transfer zone gradually will be entering into or will be occupying some portion of the active zone.

So, at any instant of time, if you find for this particular diagram has been drawn has been shown at the time instant  $t_2$ . You find that the entire bed can be divided into three regions – the equilibrium zone, mass transfer zone as well as active zone. With the progress of time, we find the equilibrium zone starts growing bigger and bigger, and the active zone starts shrinking in size.

So, therefore, as the fluid continues to flow through this particular adsorber bed. We find that the mass transfer zone also starts moving downstream in the form of a wave. The rate of movement of the mass transfer zone is usually much slower than the movement or the fluid velocity. So, the velocity of the mass transfer zone is usually much slower than the fluid velocity.

The effluent concentration from here remains almost equal to 0 as long as the mass transfer zone does not reach the effluent end. In other words, there is some amount of active zone, even if it is a very small amount there has to be some amount of active

zone after the mass transfer zone as long as that is there the effluent it comes out almost pure or almost free of the adsorbate. This can be used for any particular downstream processing.

Now, this continues as I have said as long as the mass transfer zone does not come here. If you plot the concentration of the adsorbate in the effluent concerning the concentration in the initial part. Then we find that the concentration is a very low value a permissible value which can be tolerated or which has been set by the downstream requirement of this effluent stream.

Now, as long as this continues as I have said and the empty z remains inside the bed. Then at least there is an infinitesimal amount of active zone after it. Then we have the effluent concentration to contain a very low amount of the adsorbate. Now, moment the mass transfer zone has reached the effluent end. What happens? The adsorbate starts slipping from the bed and entering the effluent.

As a result, there is an increase in the concentration of the adsorbate in the effluent stream. The first point where the effluent stream or the adsorbate concentration in the effluent stream starts increasing that particular point is known as the breakthrough point.

Normally, under ideal conditions, we moment the appearance of the adsorbate in the effluent stream is observed we call that point the breakthrough point. But usually in practical cases what happens is that we are cleaning the effluent or rather we are making the effluent free from the adsorbate so that the effluent can be used for some particular downstream processing.

So, there is some amount of a permissible limit of the adsorbate that can be tolerated in the downstream processes. Now, this particularly permissible limit or that sets the breakthrough point. The moment, we find that the concentration of the effluent has exceeded or it is approaching or it is equal to the breakthrough equal to the concentration which is permitted in the as per the downstream requirements, we identify that point as the breakthrough point.

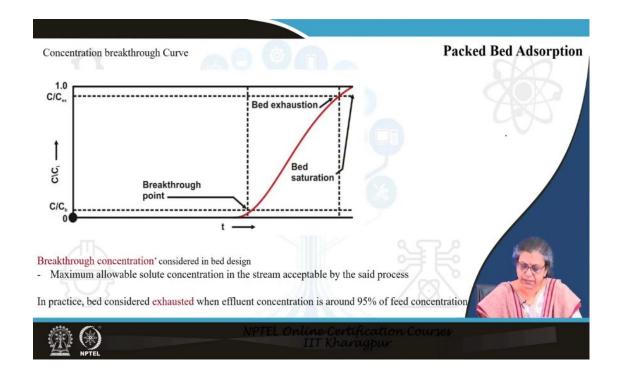
Now, if we continue the operation beyond the breakthrough point, we find that the concentration of the adsorbate in the effluent starts increasing very rapidly. Finally, it follows an s-shaped curve, and finally, the entire bed gets fully exhausted.

When the bed gets fully exhausted, under that condition, we find that the exit concentration is almost equal to the inlet concentration. Under this condition, the bed is no longer available for any further adsorption, the operation has to stop and the bed has to be sent for regeneration or desorption processes.

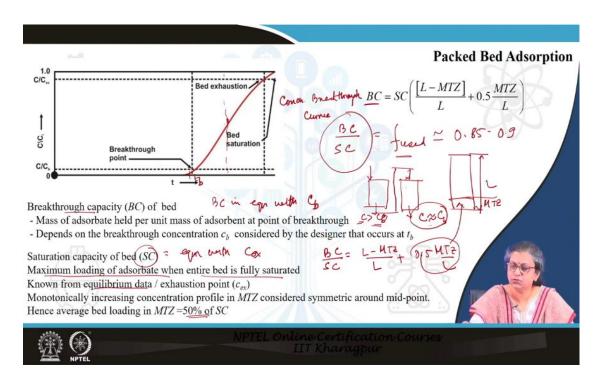
Now, this particular point, this point marks the point of bed saturation. We do not go till that point, we define a point as bed exhaustion when we find that the effluent concentration C at the exhaustion point has reached around 0.95 C<sub>i</sub>, that particular point is defined as the bed exhaustion point.

This particular and the q corresponding to this particular  $C_x$  or the q which is in equilibrium with this particular bed this particular  $C_x$ . This is known as the saturation capacity or SC.

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This is defined if you see this is defined as the maximum loading of the adsorbate when the entire bed is fully saturated. Therefore, to obtain data on SC, we need to refer to the equilibrium data or whether SC is in equilibrium with  $C_{\rm ex}$ . Similarly, if you find out that at the breakthrough point, we have reached the breakthrough capacity of the bed where again this breakthrough capacity is the mass of adsorbent held per unit mass of adsorbent at the point of breakthrough.

So, naturally, at this particular point BC, it is in equilibrium with the breakthrough concentration  $C_b$  which has been obtained at this particular point the breakthrough point  $t_b$ . So, therefore, the breakthrough capacity is the capacity of the bed or the adsorbate loading per unit mass of the adsorbent which is in equilibrium with  $C_b$  which occurs at the breakthrough point which occurs at  $t_b$ . So, therefore, this is how adsorption occurs.

Now, just as we have generated a breakthrough curve this particular curve that you see is known as a concentration breakthrough curve. It was mentioned in the previous slides. In the same way, we can also generate a temperature breakthrough curve as well for gas phase adsorption because you know one thing that adsorption is an exothermic process. There is heat released during gas or vapour adsorption from a gas mixture.

Now, normally gas has got a very low thermal conductivity, the porous adsorbent also there has gas contained, so it has a very low thermal conductivity. So, whenever heat is released the heat cannot be dissipated very fast. Therefore, this is manifested as an increase in the temperature of the effluent gas. So, what we can do? We can keep on measuring the temperature of the effluent.

We find that the temperature will be very low as long as the breakthrough point is not approached. At the breakthrough point, the temperature increases locally. By this abrupt increase in temperature, can be related to the concentration permissible or the breakthrough concentration. From this temperature rise, we can get a rough indication of the breakthrough point.

Now, normally the temperature breakthrough curve, we can generate for gas adsorption. Since in the case of liquid adsorption, the temperature rises relatively less and as the heat gets dissipated more easily. So, therefore, the temperature concentration or rather the temperature breakthrough curve cannot be generated for such a particular condition.

So, from here what do we get? We find that we can operate a bed till we reach the breakthrough point. Beyond the breakthrough point, when the bed has approached a breakthrough capacity, we cannot operate the curve operate the bed anymore because the moment we try to operate the concentration it increases very rapidly. Therefore, this cannot be permissible for the downstream equipment. Therefore, we have to stop the operation slightly before the breakthrough curve.

So, therefore, there is one other thing also that we have to remember. We must know the fraction of the breakthrough capacity as a function of the saturation capacity. Can you tell me why? Because this fraction gives you the fraction of the bed that can be utilized for this adsorption process.

Now, if this fraction is very less, then in that case it is not going to be economical for us to operate. When will the fraction be very less, you tell me? When this particular s-shaped breakthrough curve that you have gone that is going to be much more flatter.

When it is going to be much more flatter then we find that the breakthrough capacity will be a small fraction of the saturation capacity. As a result of which we find that

the fraction of the bed utilized will be very less. Definitely, we would like to maximize the amount of beds for the particular adsorption process.

So, if such a situation happens, then what do we do? Under this particular situation, we can have one particular bed where the feed enters and the effluent comes out. Now, this effluent at more or less when very less amount of bed has been utilized, this effluent it reaches the breakthrough concentration  $C_b$ . So, under that condition, what do we do? We direct this particular effluent into another bed which is placed in series with this particular bed.

Now, through this particular bed – an identical bed, we direct the effluent through this bed such that in this bed the concentration even if when it comes out from here we find that the concentration will be quite high. It may be close to the C exhaustion. Here the concentration is greater than  $C_b$ . But when it comes out from this bed, then for this bed the concentration does not exceed  $C_v$ .

So, therefore, the effluent concentration is kept within permissible limits to increase the fraction of the bed that can be utilized. What do we do? Just to optimize the two we operate a second bed in series with the first bed such that from the first bed the effluent which comes out. For that, the exit concentration is greater than the breakthrough concentration. We continue the operation such that more and more amount of the bed can be utilized.

Normally we would like to keep the fraction utilized between 0.85 to 0.9. So therefore to maintain this, the effluent concentration is greater than  $C_b$ . So, naturally, this effluent is directed through a second bed where the concentration comes to lower than  $C_b$ . Therefore, this can be used for the downstream process for which it was meant.

So, therefore, this particular ratio is very important. Normally we find that the curve is monotonically increasing in concentration profile. So, we can assume it to be symmetric about the mean axis about the centre, such that we can assume that the average bed loading in the mass transfer zone is 50 % of the saturation capacity of the bed.

If for such a situation, then we can have an analytical expression to find out the breakthrough capacity of a bed as a function of the saturation capacity. Quite naturally

in under this condition, what happens? We are having a bed of total length L out of which MTZ occurs say some particular MT some particular length say MTZ length.

So, therefore, L minus MTZ this part has already been utilized and the average concentration of this particular MTZ can be taken as 0.5 times of the SC. So, from there, we get the breakthrough capacity by the saturation capacity, which is quite naturally equal to L minus MTZ by L plus 0.5 MTZ by L, where this gives the concentration – the average concentration in the mass transfer zone, and this portion is the saturated portion.

$$\frac{BC}{MC} = \frac{L - MTZ}{L} + 0.5 \frac{MTZ}{L}$$

Now, normally, we find that if we have data on all these things. We can get an idea about the breakthrough curve from mass transfer considerations. But, usually, it is not possible. Therefore, to generate this curve and to find out how much of the fraction of the bed can be utilized. We have to again in this particular case this curve is generated on a pilot scale. With almost similar operating conditions as the actual bed.

That means the pilot bed we will be discussing more when we will be going for the bed design. The pilot bed will be operated with the same feed concentration with the same adsorbent that will be used. And will ensure that the velocity of the feed through the pilot bed is the same as the velocity of feed in the actual case such that the mass transfer coefficient is the same in the pilot bed as well as in the real the actual bed.

Based on such a condition, the breakthrough curve is generated in the pilot bed. And the information obtained from the breakthrough curve is utilized for the design of the actual bed condition.

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## **Packed Bed Adsorption**

Incorporating effect of RC (residual solute loading RC remaining in bed after regeneration), working capacity WC expressed as a measure of actual adsorption capacity of the bed.

If experimental data is available, WC may be estimated as

$$\underline{WC} = SC \left( \frac{\left[ L - MTZ \right]}{L} + 0.5 \frac{MTZ}{L} \right) - \underline{RC}$$

In absence of data WC is taken as a fraction of SC as  $WC = SC \times f_{used}$ 

Typically, 
$$f_{used} = 0.85 - 0.90$$

For design, best to determine breakthrough point and breakthrough curve for a particular system experimentally under conditions as close as possible to the process conditions.

Bed length ≥ length of MTZ ensures solute concentration in effluent stream rise from 'negligible'/ 'nil' value





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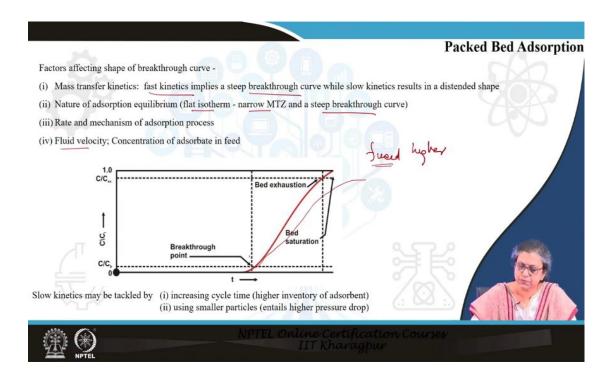
As I have said that normally these beds are hardly used as fresh beds. So, therefore, very frequently, there will be some amount of residual loading in the bed after the regeneration. So, normally to find out to estimate the working capacity of the bed, we need to consider the residual loading or in other words, we need to modify this particular expression to incorporate the residual loading.

You know if we want to find an accurate expression of the working capacity of the bed that we are going to use. In most cases, the bed does not comprise of fresh adsorbent, but of the regenerated adsorbent. So, therefore, there are two things which I would like to mention here.

First thing is that for design it's important or we should determine the breakthrough point and the breakthrough curve for the particular system experimentally under conditions as close as possible to the process conditions. Both for the actual bed as well as for the pilot scale. The bed length must be at least greater than the length of the mass transfer zone.

To ensure that, the solute concentration in the effluent is negligible, and at least to start with and the bed can be operated effectively for the specified adsorption process.

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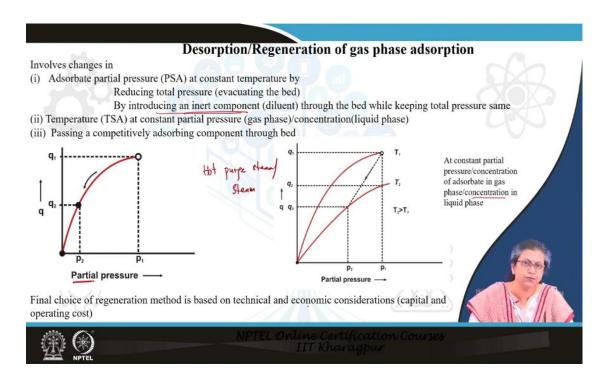
Well, this is just a recap regarding the factors which affect the shape of the breakthrough curve. Why it is important? Because we would always prefer a steeper breakthrough curve because whenever we have a steeper breakthrough curve, the fraction of the bed utilized can be higher.

So, therefore, we would always opt for a steeper breakthrough curve. Therefore, the shape of the breakthrough curve is much more important. If we have a breakthrough curve that is much more gradual, then naturally the  $f_{used}$  becomes lower. As I have already mentioned that under that condition we have to devise other ways by which  $f_{used}$  can be maximized.

So, what are the factors you tell me? If the mass transfer rate is high or the kinetics is high, under that condition we get a steeper breakthrough curve. If the adsorption equilibrium is flat, it implies that the MTZ is narrow. So, for that case also get a steeper breakthrough curve.

If the fluid velocity is less, the residence time is more. Under that condition, the mass transfer zone becomes narrower, and the breakthrough curve becomes steeper.

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Well, after we have discussed the characteristics of the packed bed adsorption, it is quite evident that for packed bed absorption, the equilibrium isotherm is important. Along with that, the breakthrough curve is also important. There is a large number of in situ factors that affect both. Therefore, the conventional practice is to generate both the curves in the pilot-scale at conditions similar to the actual plant operation and then use those things.

Now, before I go to the bed design, I thought it is very important to discuss the desorption or the regeneration of the adsorbent because mostly the regeneration part is very important. We need to regenerate the bed and use it again. Now, you tell me what are the processes or rather what can be the process for regenerating the bed.

We would first discuss the gas phase adsorption. There are since the gas phase adsorption, is influenced both by pressure and temperature. So, therefore, there are more ways of regenerating a gas phase adsorption. Then we will just be touching upon the methods of regenerating the liquid phase absorption.

Let us see for a gas phase how can we regenerate it. There are two ways. If you observe the isotherm, you will understand that to decrease the loading. We can reduce the partial pressure of the adsorbate. Once we can reduce the partial pressure, the adsorbate loading comes down or the equilibrium adsorbate loading comes down. Naturally, the remaining amount of the gas has to be dissolved from the solid. Now, for reducing the partial pressure, there can be two ways.

What are the two ways? We can reduce the total pressure. We can evacuate the bed or we can operate the bed under very low pressure. If the pressure is close to the vacuum, we call it a VSA – the Vacuum Swing Adsorption, or in other words it is called the PSA.

There is also one other way by which we can decrease the partial pressure by keeping the total pressure constant. What can we do? We can introduce some amount of inert components that also serves the same purpose and it decreases the partial pressure, accordingly, we can have desorption by PSA for gas phase adsorption.

Normally, we find that the PSAs are quite fast. Therefore, normally whenever adsorption occurs in cycles of adsorption and desorption. So, therefore, the cycle time is quite less for PSA. It is in the order of minutes. Now, other than pressure, we can also play with temperature.

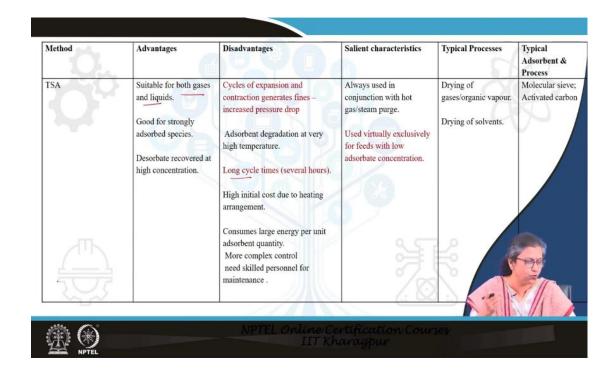
Why? Adsorption is exothermic. So, naturally, adsorption will be faster at lower temperatures and definitely adsorption operations are performed under ambient conditions. Now, suppose we heat the adsorbent bed, what happens? If we are heating it at a constant partial pressure of the gas or the constant concentration of the adsorbate in the liquid phase, naturally we find what happens that from the isotherm at  $T_1$ . It simply falls to the isotherm at  $T_2$ .

So, therefore, we can see that there is a decrease in q the adsorbate loading from  $q_1$  to  $q_2$  in this particular case. Normally, this heating is done by using some amount of purge gas or maybe by the use of hot purge gas or by the use of steam because this serves a dual purpose. What is the dual purpose? The steam firstly heats the adsorbent bed. As a result of which the temperature increases from  $T_1$  to  $T_2$ .

We find that the loading decreases along with that it also decreases the partial pressure of the adsorbate. So, naturally, we find that with this coupled effect actually. We can find that there is a decrease of q from  $q_1$  to say  $q_3$ . So, therefore, by this process adsorption becomes much more the desorption becomes much more effective.

Now, as we all know that thermal processes are always slower. Therefore, TSA requires a longer cycle time may be in terms of hours. Therefore, TSA is almost used for feeds with a very low concentration of the adsorbate.

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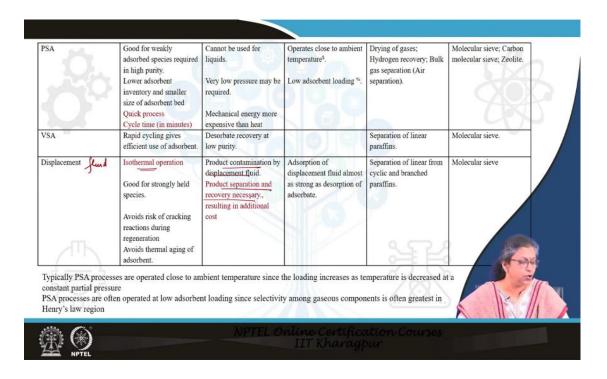


Now, other than these things I have put up a table with the different advantages, disadvantages, etcetera for TSA, PSA etcetera. So, that you can refer to this and you can find it out. TSA can be used for both liquids and gases. As I have told you that it that its main disadvantage is the long cycle time.

The other disadvantage is that when you are heating it and then you are again using it for adsorption. So, therefore, the adsorbent is repeatedly being subjected to heating and cooling. So, therefore, with this particular cycle naturally what happens, the absorbent tends to crumble up. There is a greater generation of fines.

Naturally, with that the pressure drop increases and the adsorbent also decreases with high temperature. So, these things have to be considered, but TSA is one thing which you can use both for liquids as well as gases.

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PSA, I have already defined. VSA is nothing but when the pressure is much low there is one other operation that we perform when neither PSA nor TSA is possible. What do we do under this condition? We have a bed it is saturated with adsorbed gas. We send some other either a gas or a liquid or a vapour which is known as a displacement fluid.

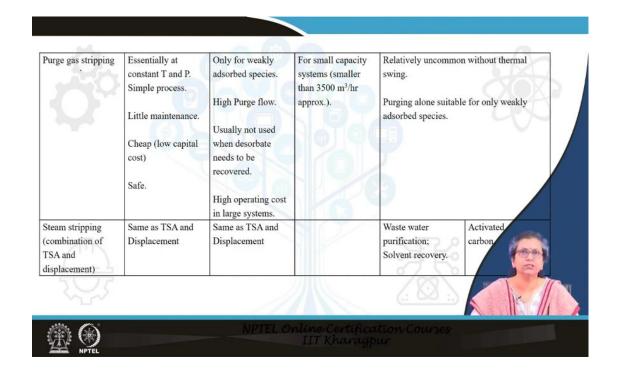
Now, what this displacement fluid does? This displacement fluid has a greater affinity for the adsorbent. Therefore, the displacement fluid tends to get adsorbed. In the process, it displaces the occluded adsorbate and releases them from the pores of the adsorbent.

Here also it serves a twofold purpose. Firstly, due to the preferential adsorption, it releases the adsorbate from the adsorbent. Secondly, it also decreases partial pressure. Therefore, it all also enhances the desorption process. The one advantage of this is that when the displacement fluid is used for the desorption process. Then two processes occur simultaneously.

Adsorption of the displacement fluid in the bed, and desorption of the adsorbate from the adsorbent. Since both adsorption and desorption occur simultaneously. So, therefore, the temperature changes, in this case, is much less. An isothermal operation can be guaranteed under or rather it is much more ensured for this particular case as well.

But along with that, there is another problem that the product it is often contaminated by the displacement fluid, and therefore, we have to take up another step where the product separation and recovery has to be considered.

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So, therefore, this cost also has to be considered when we design the adsorber bed. Now, as I have said these are almost the same as displacement fluid combined with PSA or TSA. We can use for heating we can use steam as we have said. We can use a purge gas they are almost the same.

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Sl. No.	Process Conditions	TSA		Inert Purge	Displacement	PSA	*Greater than 10:1 rat of feed to desorption
		Gas Phase Applications	Liquid Phase Applications		Only for G		pressure / vacuum desorption required
1	Liquid feed that can be completely vaporised below 200°C	Not likely	Y	Y	Y	Y	
2	Liquid feed that cannot be fully vaporised below 200°C	N	Y	N	N	N	
3	Adsorbate Concentration in feed <3 wt %	Y	Y	Y	Not likely	Not likely	
4	Adsorbate Concentration in feed 3-10 wt %	Y	N	Y	Y	Y	
5	Adsorbate Concentration in feed >10 wt %	N	N	Y	Y	Y	
6	Adsorbate recovery at high purity ≥ 90-99% rejection of carrier	Y	Y	Y	Y	Maybe <sup>&amp;</sup>	
7	Adsorbate only desorbed by TSA	Y	Y	N	N	N	all the
8	Practical (cheap, noncorrosive nontoxic adsorbate)	Y	Y	Y	Y	Y =	TO COM
9	Displacement or purge agents not easily separated from adsorbate	May be	May be	Not likely	Not likely	N	
10	Vaporised liquid / Gaseous feed	Y	N	Y	Y (.: (	Y	
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The main important techniques are first is the TSA, the next is the PSA. TSA can be used for both liquid and gas desorption. It is generally slower and there are some problems related to thermal ageing the thermal deterioration of the adsorbent. PSA can be is used very effectively for gas-phase adsorptions, and it is much faster also. The displacement fluid technique that is used when usually when we want an isothermal operation and both PSA and TSA can be are not very favourable right.

We are going to discuss the design in the next class. And I like to mention that there are there is a rigorous method that involves mass momentum, energy balance, and equilibrium relationships. But often this does not necessarily lead to an accurate result just because of the unavailability of data.

Therefore, an empirical approach is usually used based on data or from the pilot plant. We are going to discuss that in detail in the next class. Then also we will be working out a problem to illustrate the design principles that will be discussed. So, with this, I thank you all.

Thank you very much.