Physico-Chemical Processes for Wastewater Treatment
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Lecture 32
Adsorption II

Good day everyone and welcome to these lectures on Physico-Chemical Processes for Wastewater Treatment. In the previous lecture, we started with one of the unit operations which is called as adsorption and which is most commonly used in all the water treatment and wastewater treatment processes, it may be used in the initial stages and it may be used in the tertiary stages in the wastewater treatment. So, both are possible.

Now, continuing with the previous lectures. In the previous lectures we studied that there are 3 important properties which are the most important characteristics which are desirable in any adsorbent.

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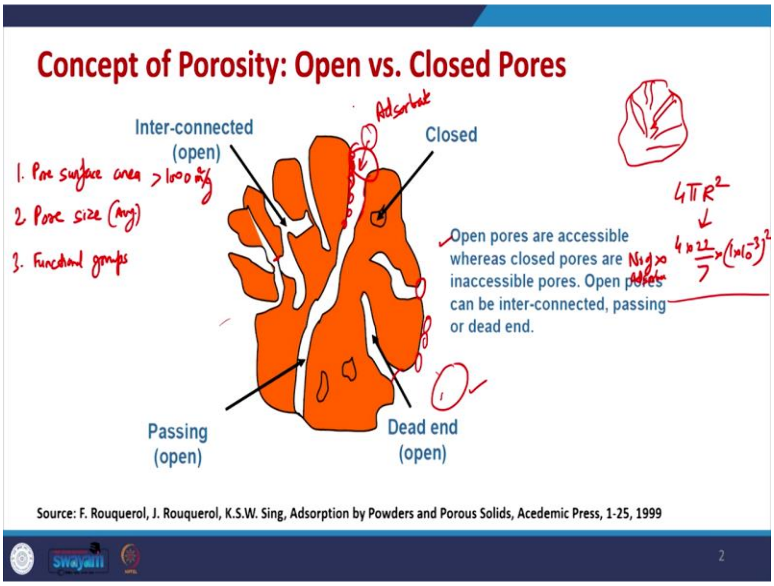

Concept of Porosity: Open vs. Closed Pores

Open pores are accessible whereas closed pores are inaccessible pores. Open pores can be inter-connected, passing or dead end.

Source: F. Rouquerol, J. Rouquerol, K.S.W. Sing, Adsorption by Powders and Porous Solids, Acedemic Press, 1-25, 1999

So, these properties included the surface area or pore surface area which was the first property. Second was the what is the pore size of the, average pore size to that is very important property and the third property which was there, what are the functional groups which are present on the surface of the adsorbents, so this was there.

Now, the pore surface area and pore size that 2 properties tell us that there are 2 things, if suppose any adsorbent is there, so, that adsorbent maybe having different pores the way it is shown here in this slide. So, its outer surface area will be different. And suppose the any spherical particle is there, suppose this adsorbent is spherical in nature.

So, its outer surface area is 4 pi R square, so, that we know very well where R is the radius, so this is known. But remember in the previous lecture, we studied the surface area is in the range of 4 pi like 1200 or 1000 meter square per gram or more, so for activated carbon, so, commercial activated carbons, so surface area maybe more than that. So, if we consider 4 pi R square, the external surface area will always be less. So, this is there, so, 4 into 22.7 and suppose the diameter of the particle is very less. So, depending upon that is suppose 1 millimeter size is there also.

So, for 1 millimeter 1 into 10 raise to minus 3 square and in 1 gram suppose, we may have tentatively some number of adsorbents, so, in 1 gram number of adsorbents, may be selected number of particles of the adsorbent that may be there. So, if you multiply this external surface area will be much, much, much lower than the pore surface area which is there as well. So, that means, what we require is that adsorbent should be highly porous with lots of pores, in which the adsorbent or the adsorbate may move. So, what we desire is that, the adsorbate should be able to move inside these pores and this should be highly porous.

So, that the surface area this surface area which is there in the pore that is available for adsorption to occur, where the molecule adsorbate molecule may come, they may like get a absorb here. So, we always require highly porous adsorbent. Some of the pores may be open some of the pores may be closed. So, there are different types of pores which are possible, so, that we will study more. But here we can see here, open pores are those which are accessible to the adsorbate, whereas the close are those which are inaccessible, so, open pores can be interconnected they may have a passing connection also.

They may be totally blocked also they may be dead end. So, like here you can see interconnected pore. So, this pore is there is suppose this is open so, this is also interconnected. So, this interconnectivity may be there, this is passing pores. It is all throughout the adsorbent itself. So, this is like a passing pore which is there, this pore is dead end for because it closes at one end. So, there are different types of pores possible.

Now, these pores can also be classified for the like cylindrical pores, etcetera. So, we will discuss those also in later on. So, this is, so, important thing is that what is the average pore size which is there, so, can any adsorbent suppose the adsorbent molecule is this. So, these are adsorbent molecule despite this adsorbent having a lot of pores, this cannot go inside any of the pores. So that means this adsorbent will be having very, very less capacity with respect to adsorption of this particular adsorbate and this adsorbate which may not get inside these adsorbents, so it may maximum get connected at the surface only.

So, its adsorption capacity will be very, very less. So this is there. Going further, there are many steps which occur during the movement of this adsorbate inside these pores until unless they get adsorbed. So, what are those steps?

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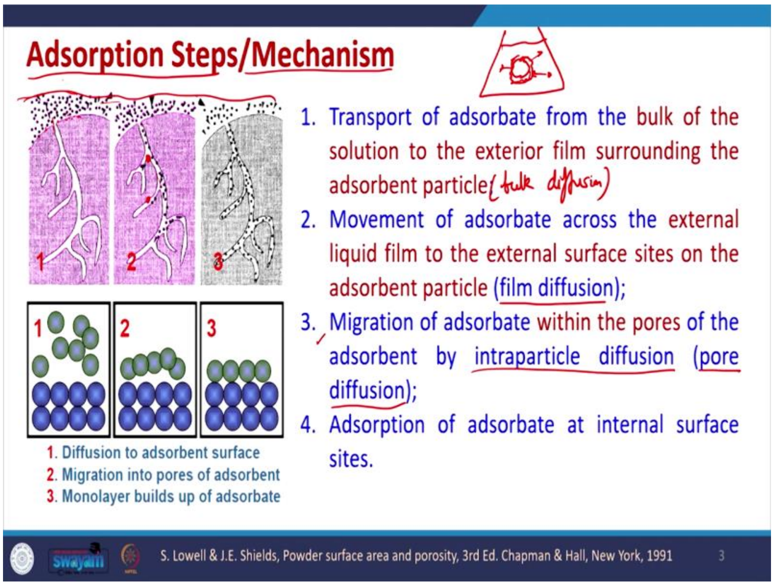

Adsorption Steps/Mechanism

1. Transport of adsorbate from the bulk of the solution to the exterior film surrounding the adsorbent particle(bulk diffusion)
2. Movement of adsorbate across the external liquid film to the external surface sites on the adsorbent particle (film diffusion);
3. Migration of adsorbate within the pores of the adsorbent by intraparticle diffusion (pore diffusion);
4. Adsorption of adsorbate at internal surface sites.

1. Diffusion to adsorbent surface
2. Migration into pores of adsorbent
3. Monolayer builds up of adsorbate

S. Lowell & J.E. Shields, Powder surface area and porosity, 3rd Ed. Chapman & Hall, New York, 1991

So, for understanding this, we must again go back and try to understand the well in the previous slide like in the previous lecture we discussed suppose, there is a this is a beaker is there and beaker is having this solution which contains the adsorbate which has to be adsorbed. Now, it contains an adsorbent particle and these adsorbent particle is there and this is some molecule which is to be adsorbed. So, there are different sections of movement of this particular adsorbate it inside this adsorbent.

So, these are called as adsorption steps or the mechanism how which this adsorbate is moving inside this adsorbent and getting adsorbed. So, this is the, so, the first phase is transport of adsorbate from the bulk of the solution to the exterior film surrounding the adsorbent particle. Now, whenever there is adsorbent particle in the liquid phase, it will always be surrounded by some liquid film, which will always move along with this adsorbent wherever it moves.

So, that is there. So, liquid film a stagnant liquid film will always be there around the adsorbent molecule. So, that means the adsorbate has to first move to the exterior film surrounding the adsorbent particle. So, this is the first step. Now, second step is that movement of adsorbate across the external liquid film to the external surface sight on that jar adsorbate particle. So, that is it the adsorbate it has to move across the film. So, this is called as film diffusion and so, and first phase it is called as also called as bulk diffusion also. So, this is like bulk diffusion.

Now, in the third stage, migration will occur within the pores of the adsorbent. So, suppose this is the film you can see here the film is there, now, the adsorbent will move inside the pores that means, we can see that adsorbent has move up to the surface, it has come inside the surface but it has not moved inside the pores. Now, it will move inside the pores, so migration of adsorbate within the pores of the adsorbent by intraparticle diffusion, it is also called as pore diffusion and pore diffusion may also be, further be classified like whether it is moving from bigger size pores or smaller size pores. So, that is also possible.

Now, when the diffusion will occur after diffusion occurring, then the adsorbate will get adsorbed at some surface site. So, suppose surface site is here or here also. So, first molecule will come and get adsorbed here. So, this is the last step or 4th step adsorption of adsorbate at the internal surface sight. So, this is the step.

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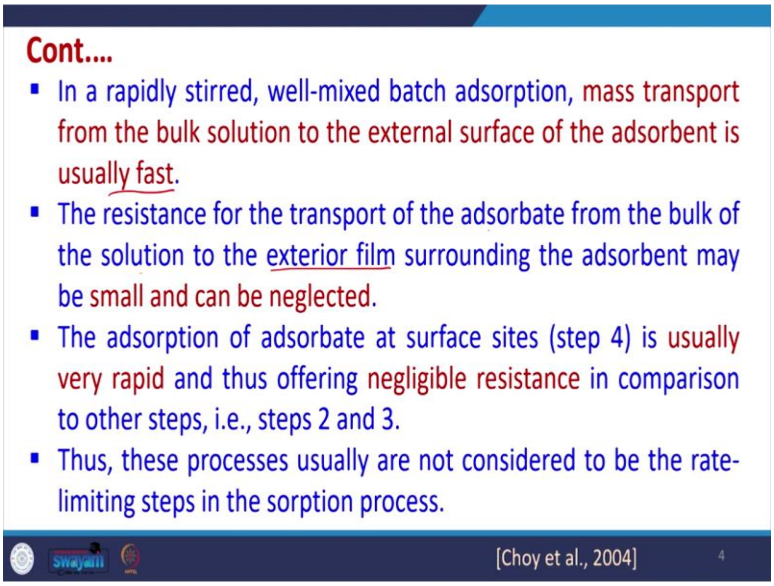

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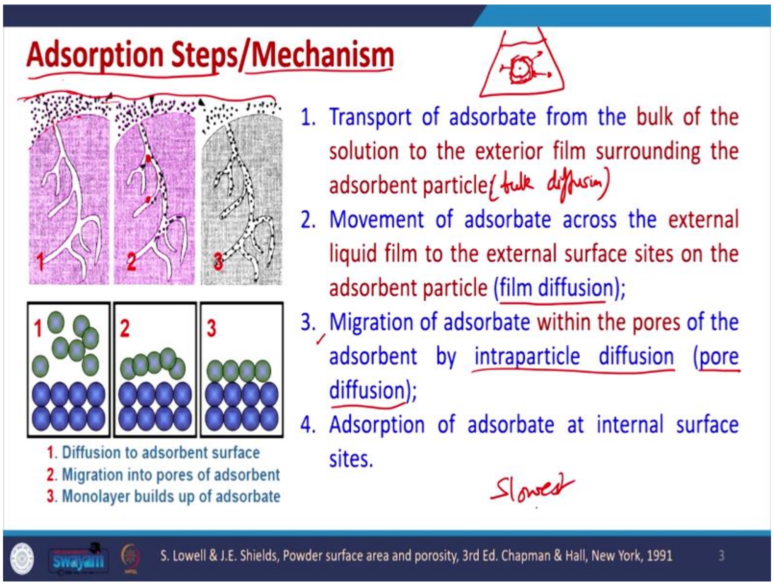
- In a rapidly stirred, well-mixed batch adsorption, mass transport from the bulk solution to the external surface of the adsorbent is usually fast.
- The resistance for the transport of the adsorbate from the bulk of the solution to the exterior film surrounding the adsorbent may be small and can be neglected.
- The adsorption of adsorbate at surface sites (step 4) is usually very rapid and thus offering negligible resistance in comparison to other steps, i.e., steps 2 and 3.
- Thus, these processes usually are not considered to be the rate-limiting steps in the sorption process.

[Choy et al., 2004]



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Slowest

S. Lowell & J.E. Shields, Powder surface area and porosity, 3rd Ed. Chapman & Hall, New York, 1991

Now, going further now, if these 4 steps are there suppose, so, this is like a adsorbate moving from the bulk of the solution and going inside this particle adsorbing particle. Now, we want all these steps to be as quick as possible, because overall contact time, the time which will be there for adsorption to occur will be dependent whether how fast are these steps are. So, for doing this, we need to fasten all these processes. So, that we can identify the rate controlling step are the slowest step and we always try to see that the slowest step also becomes faster.

So, that the overall reduction in the adsorption time occur. So, if this is possible, we can have a packed bed which can take more amount of material within the same time. So, all those conditions we always want that kinetics to be faster or the adsorption process to occur very quick. So, that means we have to see that these steps which are their bulk diffusion, film diffusion, pore diffusion and ultimately the adsorption occurs very quickly. So, for doing this now, suppose the in a rapidly stirred well mixed batch system, suppose this is only a batch stirrer is there.

So, under that condition, since, it is very well mixed the mass transport from the bulk solution to the external surface film of the external surface film of the adsorbent will be very fast that means, the bulk solution is always good, if we are rapidly stirring the solution, so that problem is not there. Now, for the second case, the resistance for the transport of the adsorbate from the bulk of the solution to the exterior film surrounding the adsorbent may be small, if it is very fast and that can be neglected. So, bulk diffusion is not an issue, if we have a rapidly stirred system is there.

Now, the adsorption of the adsorbate at the surface site that means, the fourth step, this step is also very quick generally it is very, very quick and it has negligible resistance as in comparison to steps 2 and 3. So, first step bulk diffusion and the fourth step adoption they are always very quick. So, there is not much of an issue, the issue generally in lie with film diffusion or the pore diffusion that means, they will be generally the slow system. Now, if you have a rapidly mixed batch system, the film division will also be the film will be lesser in its thickness will be less.

So, that means the molecule has to only diffuse a very small distance. So, that means the film diffusion will also be faster as compared to the pore diffusion. So, under that condition, the pore diffusion may be the rate controlling each step. So, either secondary step or third step they may be rate controlling. Now, in most cases steps second and third, which are like sorption phenomena, they control the sorption phenomena and they are like film diffusion and pore diffusion. So, there are different possibilities which are there with respect to these 2 diffusions.

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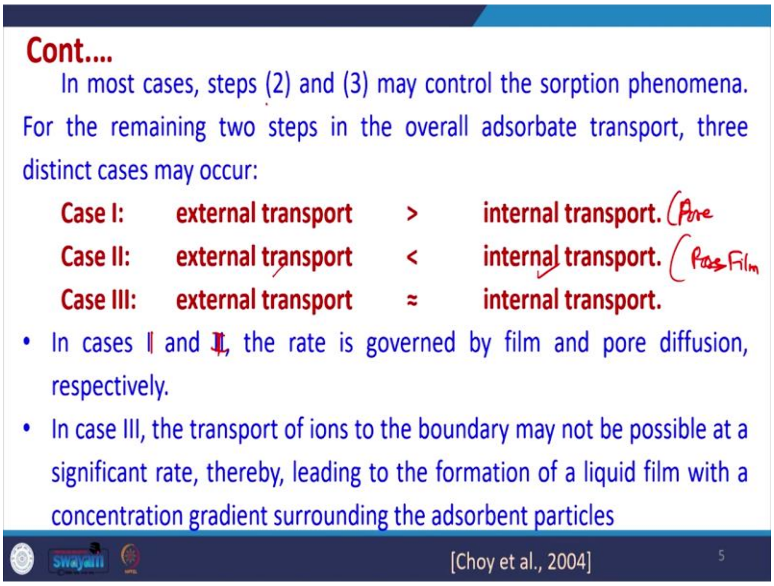

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In most cases, steps (2) and (3) may control the sorption phenomena. For the remaining two steps in the overall adsorbate transport, three distinct cases may occur:

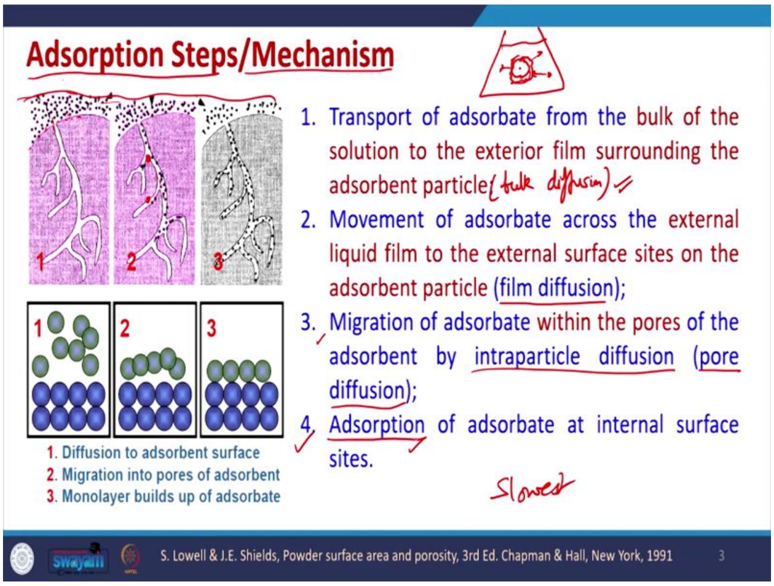
Case I: external transport > internal transport. (Pore

Case II: external transport < internal transport. (~~Pore~~ Film

Case III: external transport ≈ internal transport.

- In cases I and II, the rate is governed by film and pore diffusion, respectively.
- In case III, the transport of ions to the boundary may not be possible at a significant rate, thereby, leading to the formation of a liquid film with a concentration gradient surrounding the adsorbent particles

[Choy et al., 2004]



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So, in the case when the external transport may be faster that means the film diffusion is faster as compared to the pore diffusion, second case the external transport is lower as compared to internal transport. So, that is possible so pore diffusion may be the rate controlling step and in some of the in some cases both are equally similar to the condition. So, it is possible. So, here the read in the first step it is controlled by film diffusion, here external transport is fast for it is pore diffusion limited. Here it is film diffusion limited because here the internal transport is very quick and external transport is slow.

So, it is opposite way it is written it is first and second and first it should be written. In the case third, the transport of ions to the boundary layer may not be possible at a significant rate and thereby leading to the formation of a liquid film with a concentration gradient surrounding the adsorbent particle. So, it is possible so, we will have to see and examine and properly optimize these conditions. So, that at least if you have proper selection of adsorbent and we have conditions which are good enough, we can avoid these slower steps also or minimize the resistances which are there.

So, these resistances we need to determine and we need to understand, so, that we can properly select an adsorbent and our overall treatment process will become faster because of that, so this is there. Now in the fourth step which occurs where the adsorption occurs, this adsorption of adsorbate at the internal surface site that may occur by 2 mechanisms.

One is that there is some chemical bonding occurring that the surface site contains some functional groups which is polar, that adsorbate is also polar and they are of opposite nature. So, they will attract each other and adsorbed chemical bonding may occur. Similarly, if it is not there, both are non-polar. So, under that condition some formation or some physical attraction may occur and that will be governed by van der Waals forces of attraction. So, this is possible. So, under that condition also, we have van der Waals forces of attraction. So, chemisorption and physisorption are the actual terms which are used for finding out the last step by which that reaction may occur.

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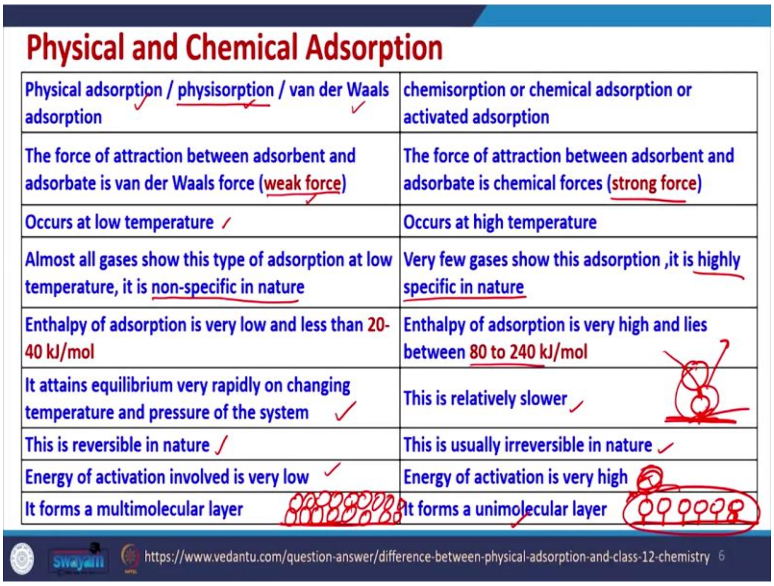
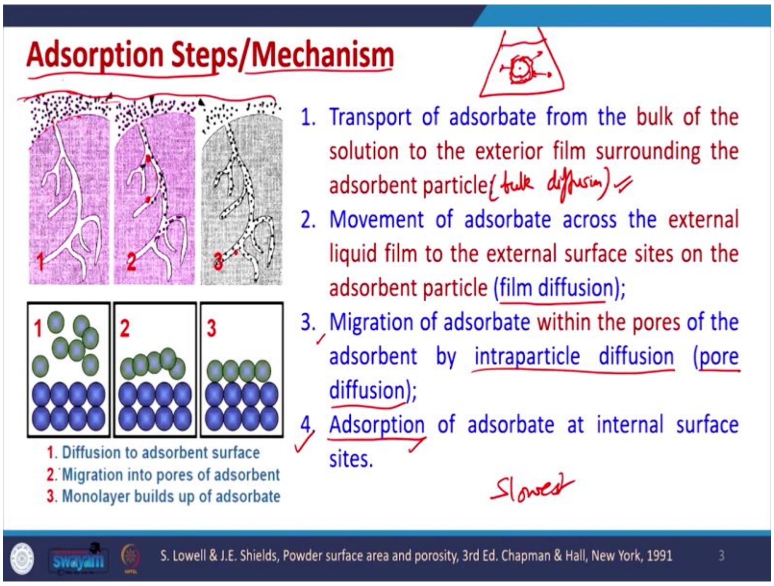


Physical and Chemical Adsorption

Physical adsorption / physisorption / van der Waals adsorption	chemisorption or chemical adsorption or activated adsorption
The force of attraction between adsorbent and adsorbate is van der Waals force (weak force)	The force of attraction between adsorbent and adsorbate is chemical forces (strong force)
Occurs at low temperature	Occurs at high temperature
Almost all gases show this type of adsorption at low temperature, it is non-specific in nature	Very few gases show this adsorption ,it is highly specific in nature
Enthalpy of adsorption is very low and less than 20-40 kJ/mol	Enthalpy of adsorption is very high and lies between 80 to 240 kJ/mol
It attains equilibrium very rapidly on changing temperature and pressure of the system	This is relatively slower
This is reversible in nature	This is usually irreversible in nature
Energy of activation involved is very low	Energy of activation is very high
It forms a multimolecular layer	It forms a unimolecular layer

https://www.vedantu.com/question-answer/difference-between-physical-adsorption-and-class-12-chemistry



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So, in the physical adsorption, which is also called as physisorption or van der Waals adsorption, this is one of the method of adsorption in the fourth step. Now, similarly, chemisorption, chemical adsorption or activated adsorption this is also possible now, both have different mechanisms and different features which are compared here. Now, the van der Waals force of attraction is a weaker force that means, the force of attraction between adsorbent and the adsorbate is van der Waals forces and it is weaker in the physisorption which is called a physical adoption.

If the force is weaker, so, at least there is a possibility that adsorption may occur, but suppose later on when the all the adsorbent has been totally spent, that means, there is no further capacity. So, we can take out the adsorbent and we can dissolve the adsorbate out of the adsorbent. So, it is possible to regenerate the adsorbent more easily in the physisorption as compared to in the chemisorption. Because in the chemisorption, we have the force of attraction between the adsorbent and the adsorbate by chemical forces that means some bonding may occur.

So, these bonding may be strong. So, that is if we have to regenerate the adsorbent it is not possible as easy it is in the physical adsorption. So, this is one difference. Second, there is another difference that generally physisorption occurs at low temperature whereas chemisorption occurs at high temperature. So, this is possible. So, this is general it may be possible vice versa also, but this is the general difference, which is there.

Almost all gases show physisorption at low temperature and it is nonspecific in nature, nonspecific in nature means, that any type of gas it may get physically attracted and get adsorbed. Whereas, in the chemisorption, some specific nature of adsorption may occur, because the gas may be inherently acidic in nature and the adsorbent may be basic in nature. So, under those conditions that chemisorption may occur and but it will be not nonspecific. Why this is so, because it will only adsorb those gases which are acidic because the adsorbent itself is basic in nature.

So, it will not absorb anything which is basic. So, that means, the adsorption will be only highly specific in nature. So, this is possible. Now, enthalpy of adsorption for the gas phase separation is like for physisorption it is lower 20 to 40 kilojoules per mole, it is much, much higher for chemisorption it is given here. Now, the physisorption attains very quick, very rapid and on change in temperature and pressure of the systems, so this is faster as physisorption is faster as compared to chemisorption which is slower this is reversible in nature already showed because it is weak force of attraction.

So, we can easily apply some other force or temperature to dissolve the gases or the any pollutant out of that. So, it is reversible in nature, and this is not reversible. A chemisorption is not reversible, because chemical bonding has occurred. So, suppose we have to take out or it is possible that we may have to use some solution or something which may have a stronger bonding towards the adsorbate as compared to the adsorbent itself. So, that means suppose this is the adsorbent surface. And now this adsorbate has come and there is a chemical bond.

Now suppose now, what we want? We want to take out this. So, that means there should be another molecule which should come and it should have more stronger bond then this bond then only this bond can be broken, otherwise, this irreversibility will not cannot be changed, and it will always this will be always be attracted towards the surface, so this is there and because of that the these things the energy of activation involved in the physisorption is low and it is very high in the case of chemisorption.

Now, since, and this is the last difference, which is there, it is very, very important, since the physical nature of adsorption is there, it is possible to have multi molecular levels layers also, because, suppose the surface is there. So, this is a surface and one molecule may will come and they will bond like this. So, this physical adsorption has occurred.

Now, if suppose the surface gets saturated, so, another layer may get formed because it is only physical nature of adsorption. So, bonding may occur between the adsorbate adsorbate also so, multiple layers are possible, this is not possible in the case of chemical option, because some bond formation has happened between the adsorbent and the adsorbate. So, and that bond formation is not possible within the adsorbate now, this bond is not possible to be found within the adsorbate adsorbate.

So, that is why in chemisorption only single layer adsorption may occur, if any multi-layer formation is occurring, so, that will be only physical in nature. So, (()) (23:00) the other layers which may get formed that will be more physical adsorption after one layer which has been formed because of the chemisorption. So, these are the various differences which are there between different types of attraction or the forces which may be there, while the adsorbate gets adsorbed on the surface of the adsorbent.

So, we can see here, so, the diffusion is occurring in this first step then here migration into the pores is happening and then ultimately mono layer buildup is here this is the mono layer buildup which has occurred. So, mono layer is more possible in the case of chemisorption and multi-layer is more possible in the case of physical adsorption or physisorption. Now, there are various factors which control the adsorption process. So, what are those some of these things we will try to discuss today.

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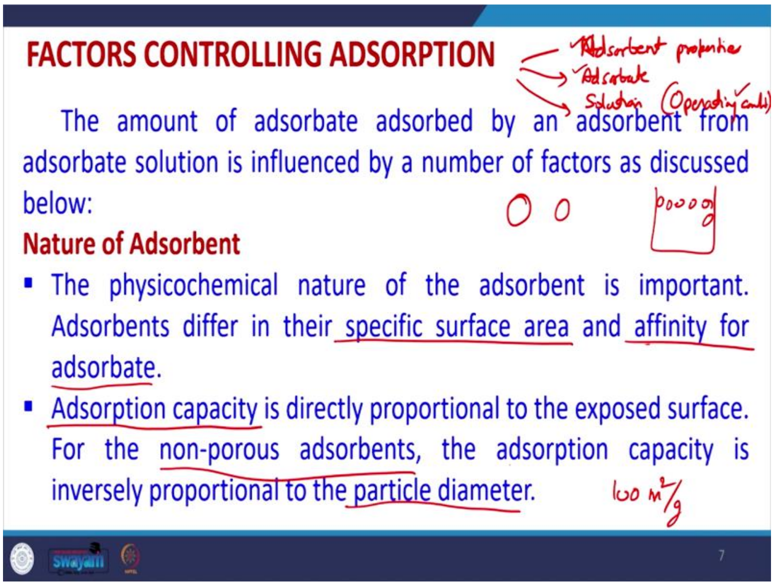

FACTORS CONTROLLING ADSORPTION

The amount of adsorbate adsorbed by an adsorbent from adsorbate solution is influenced by a number of factors as discussed below:

Nature of Adsorbent

- The physicochemical nature of the adsorbent is important. Adsorbents differ in their specific surface area and affinity for adsorbate.
- Adsorption capacity is directly proportional to the exposed surface. For the non-porous adsorbents, the adsorption capacity is inversely proportional to the particle diameter.

So, already we have discussed one of the important parameters with respect to now, the factors which may control the adsorption, they may be classified into 3 categories in a way. One is like dependent upon the adsorbent. So, this is possible the property of the adsorbent controls the adsorption phenomena, so, adsorbent properties become very important or nature of the adsorbent that becomes very important. Second thing is the properties of adsorbate that may also be crucial in deciding whether the adsorption may occur on may not occur.

And third property is the solution itself, whether it is acidic, basic, or whether it is gas phase-liquid phase, etc. So, all these factors may affect within the solution or within the treatment operating conditions may also become very important with respect to controlling the adsorption. It is possible to change the operating parameters like temperature and by doing this at one temperature there is it is possible that there is no adsorption occurring, but higher temperature or lower temperature adsorption may occur significantly.

So, we can always manipulate with the operating conditions later on. But first and foremost adsorbent properties and the nature of the adsorbate and the solution. They become very, very important. Now, we will start with the nature of the adsorbent. Now, already we have studied discussed few of the properties with respect to nature of adsorbent and the Physico-chemical nature of the adsorbent is very important and adsorbents, they may differ in the specific surface area and affinity for the adsorbate.

Now, a specific surface area may change, many commercial adsorbents may have very high surface area like metal organic frameworks may have more than 5000 meters square per gram up to 78000 also they have gone. Now, there are some simple charcoal that may be having only surface area of 100 meters square per gram, but that will be available very, very cheaply. So, this is possible. Now, affinity may also be different for different types of adsorbents, already we have discussed whether adsorbate is polar, non-polar, whether it is acidic or basic.

So, all these properties affect its affinity towards the adsorbent. So, adsorbent affinity or adsorbents property also become very important. Now, adsorption capacity that we have earlier discussed is directly proportional to the exposed surface area. So, that means, for the non-porous adsorbent suppose, it has very, very less surface area around 10 meters square per gram. So, under that condition suppose, we have 2 adsorbents both are having same capacity or they have very less capacity. So, they that will depend upon the for nonporous adsorption the adsorption capacity depends upon the particle diameter.

So, if is there is 2 balls which are there. So, smaller size balls will be having higher surface area and we can keep more number of balls within a certain volume. So, that is possible so, we have more external surface area. So, for non-porous adsorption, the adsorption capacity is inversely proportional to particle diameter. Now, for porous materials, the material highly porous it has very high surface area etcetera under that condition, it will be independent of the particle size. So, that the adsorption capacity will not depend upon the particle size. Particle size will still be important with respect to pressure drop and other things.

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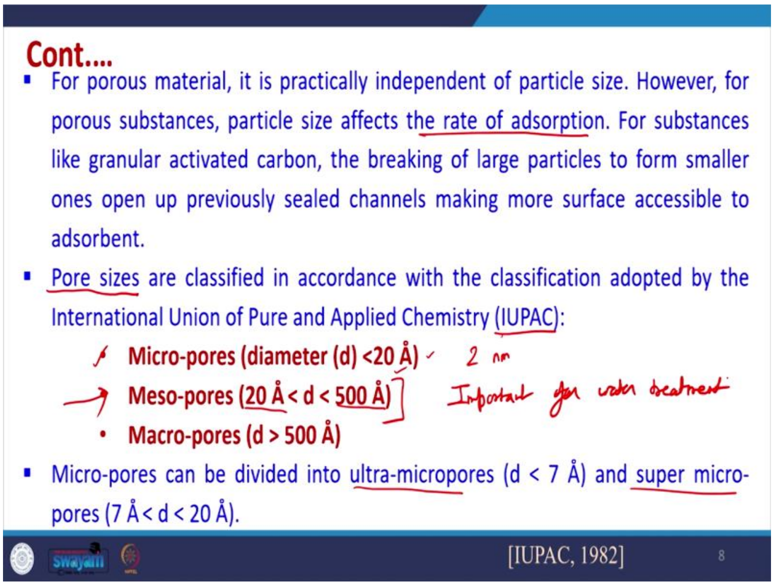

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- For porous material, it is practically independent of particle size. However, for porous substances, particle size affects the rate of adsorption. For substances like granular activated carbon, the breaking of large particles to form smaller ones open up previously sealed channels making more surface accessible to adsorbent.
- Pore sizes are classified in accordance with the classification adopted by the International Union of Pure and Applied Chemistry (IUPAC):
  - **Micro-pores (diameter (d) <20 Å)** ✓ 2 nm
  - **Meso-pores (20 Å < d < 500 Å)** Important for water treatment
  - **Macro-pores (d > 500 Å)**
- Micro-pores can be divided into ultra-micropores (d < 7 Å) and super micro-pores (7 Å < d < 20 Å).

[IUPAC, 1982]

However, for porous substances the particle size affects the rate of adsorption, it is possible. So, this is there. So, that that is different thing the adsorption capacity we may have different.

For substances like granular activated carbon, the breaking of large particles to form smaller ones, open up the various sealed pore or channels making more surface accessible to the adsorbent. There is another very important property which was highlighted in previous slide is pore size.

So, there are classification of pore sizes. So, as per International Union of Pure and Applied Chemistry, IUPAC, there are 3 types of pores which are classified. So, one is like micro pores, which are having diameter less than 20 angstrom, so this is there, that means, 2 nanometers, so, sometimes in the nanometer it is also written.

Similarly, meso-pores have diameter in the range of 20 angstrom to 500 angstrom and micro pores are those pores which are having diameter more than 500 angstrom. Now, micro pores can also be further divided into ultra-micro pores and super micro pores. So, ultra-micro pores are having diameter less than 7 angstrom and super micro pores have diameter between 7 to 20 angstroms, so it is possible.

Now, for gas phase separation, micro pores are important because even more micro pores are there within the same surface area the surface more of the pores can be utilized for gas phase separation, but for liquid phase adsorption for water or wastewater treatment meso-pores are very important for water treatment. So, we always look for adsorbent which are more meso-pores. So, this is very important. So, this is more important for water treatment as compared to any other thing. So, meso-pores is the pores which are we are looking for water or wastewater treatment.

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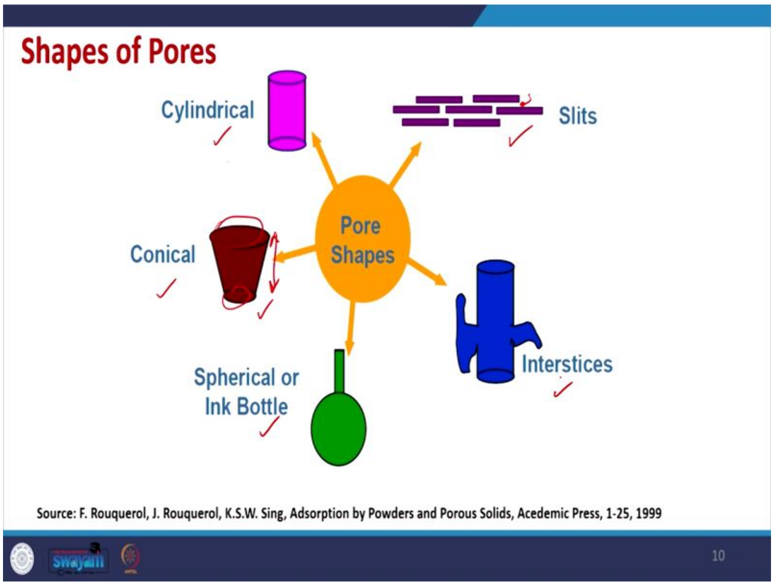

Shapes of Pores

Source: F. Rouquerol, J. Rouquerol, K.S.W. Sing, Adsorption by Powders and Porous Solids, Acedemic Press, 1-25, 1999

Because generally the pollutants which are present in the liquid steam are always half diameter bigger size as compare to 2 nanometer. So, we have mesoporous silica, activated carbons, all these things can they can be used more because they have more surface pore size, which is between 2 nanometer to 50 nanometer or 20 angstrom to 500 angstrom which are listed here.

Now, micro pores which are used for gas phase separation generally we always see that zeolites, metal organic, framework, activated carbons also can be used, because activated carbon may have micro pores also. For mesopores is more true for water treatment or wastewater treatment. So, we always look for mesoporic adsorbents, which may have pores more in the mesoporous rain for water treatment, this is used.

Micro pores are not that much considered, because if any material is having more macro pores it may be underutilized. So, micro pores are only bigger sized pores where the initial transport of the adsorbate will occur, so this is there. Now, that there are different possibilities of shapes of pores also.

Already we discuss that there is a possibility of cylindrical pores like here you can see we can we consider this to be a cylindrical pore. So, this is possible cylindrical pores are possible, there are many other types of pores are also possible like slit. So, this is possible. So, it may be having slit shaped pores, it may be having conical pores initially the pore size is bigger, but it is becoming smaller and that length in which it is becoming smaller is very small that will fall into the category of conical pores there is a possibility of ink bottle type of pores.

So, that means, the inside the pore is very big, but the path which is there for moving inside is very small. So, that will come under the category of ink bottle pores. So, this is possible. Similarly, in between interstices may be there. So, these type of pore shapes are also possible. So, we always tried to analyze all these types of pores, etcetera. And we try to analyze all these things and we tried to discuss the, we can determine the pore shapes, pore surface area and what is the pore size through a method which is called as liquid phase nitrogen adsorption.

So, what we do is that we adsorb the liquid at very low temperature liquid, we absorb the nitrogen at very low temperature and that temperature is less than minus 193 kelvin, so, sorry less than minus 193 degrees centigrade. So, under those conditions, we try to find out that how much liquid nitrogen has been adsorbed and through the different shapes of the adsorption isotherm which are there different types of classification of pores, pore diameter, pore surface area, everything can be determined.

So, we will discuss all these things further in later classes. And we will understand the other factors also which affect the adsorption process along with the adsorbent properties. So, we will discuss all those properties in the next lecture and what are the different parameters which affect the adsorption process. Thank you very much.