

Chemical Reaction Engineering
Prof. Jayant Modak
Department of Chemical Engineering
Indian Institute of Science, Bangalore

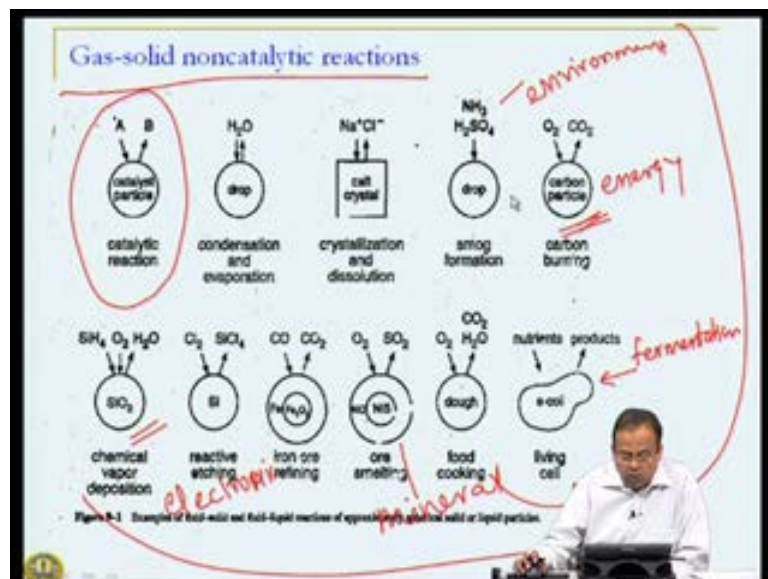
Module No. #04

Lecture No. #24

Gas-Solid Non-catalytic Reactions

Friends! Let us start discussion on a new topic in this session and today, we will focus on gas-solid non-catalytic reactions. Just to recap what we have seen in several previous sessions, we looked at gas-solid catalytic reactions. Now, when it comes to non-catalytic reactions, there are several features of gas-solid catalytic reactions that get carried over; however, there are certain new aspects that need to be considered and therefore, we will focus on those new aspects and things like diffusion in the solid or multi-component diffusion, effective diffusivity, all those concepts from gas-solid reactions will now be taken. We know what these are. Therefore, I would not be spending much time on these aspects.

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So, let us start with looking at few examples, where we encounter gas-solid Non-catalytic reactions. So, what you see here is a host of examples, starting with the first one of course, it is the best case as you may want to call it, that is a catalytic reaction which we have seen so far. So, where you have a catalyst, you have a reactant which comes to the catalyst surface, diffuses inside and we have a reaction taking place. Few examples which involve both solid as well as liquid particle and the gas phase are depicted in this particular slide.

Starting with simple process of condensation and evaporation of a drop which involves water in a vapor form either condensing to become a liquid drop or a liquid drop evaporating, we have crystallization and dissolution. Let us say, we have a salt crystal and it dissolves in water. Then, we have little more complicated process, something which we are all worried about. There are a whole lot of international conference agencies going on environment and one part of this environment is a smog formation. We have a drop of ammonia or Sulphuric acid which combines with the water and we have a smog formation. Something which gives us energy that is cold thermal power plants for example, this process involves a carbon particle which is oxidized by oxygen either in the air or in the pure form, very rarely you will use oxygen in the pure form, but this carbon particle now burns to give rise to CO_2 , if it is a clean burning. If it is a partial oxidation, then, you have lot of obnoxious gases such as carbon monoxide and so on. Then, we have an example from semiconductor industry. For example, chemical vapor deposition, we have silica or we want to make silicon dioxide substrates for making some chips or whatever it is, **we** form that with gas silicon tetra hydride and oxygen depositing on to a **solid** surface and giving rise to SiO_2 . Sometimes, if you have seen an IC circuit, you will find there are a lot of grooves and other things. So, you want to etch out from an already available semiconductor surface. So, this etching process is also gas-solid non-catalytic reaction. For example, you treat it with chlorine, you get silicon tetrachloride which is a vapor, chlorine is a vapor, silicon is a solid. So, there is a reaction that is taking place in which material is **getting** etched out.

Then, we have iron ore refining. For example, you want to make iron from ore such as hematite, then we have the iron ore, you treat it with carbon monoxide, reduce that Fe_2O_3 to Fe, carbon dioxide is released in the process. So, again it is gas-solid non-catalytic reaction. Then, you could have ore smelting sulphides of various different

metals in the form of minerals or ores giving rise to oxides. For example, nickel sulphide giving rise to **nickel** oxide.

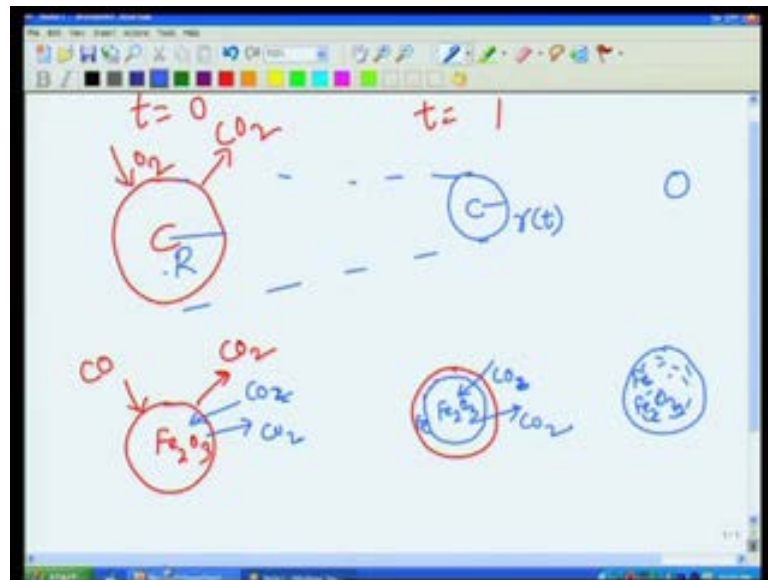
Food for thought: you know that you enjoy nice fluff breads or rotis or naans and so on. How are these made? These are made from solid or semi-solid dough and **you** use some livening agent or yeast for example, which takes the oxygen, carbons it to carbon dioxide, in the process, the bubbles that pass through this dough gives it that fluffy or **spongy** nature and an ultimate example, if you want to call, it is a living cell which is actually not a solid. It is neither liquid. It is a membrane bound liquid material, if you like to call it in a simplistic term, where it takes up the nutrients, make **some** products. So, these are large number of examples which refer to gas-solid non-catalytic reactions.

Compared to catalytic reactions, there are several features which are common as I said, but there are several features which are also not seen in gas-solid catalytic reaction, but before we venture into those, I want you to look at this example in a **slightly** different way. What makes chemical engineers dive whole heartedly into variety of **different** fields? As I have said in my introductory remarks for this course, we as chemical engineers, we do not focus on the product, but rather, we focus on the process and this particular slide that you are seeing is a classic illustration of that. By focusing not on burning of carbon or dissolution of crystal, but rather, the processes which drive **these** products mainly in this particular case, gas-solid non-catalytic reactions, its kinetics, its diffusion and so on. We can apply the same principles to variety of different sectors. For example, we have energy here, in the form of coal burning. We have environment here in the formation of smog and so on. We have entire fermentation industry and bio industry over here. We have food industry covered. We have mineral processing covered. We have electronic industry covered.

Had we focused on individual products such as silicon chips or iron ores or living systems, we would have missed out the broad principles which guide all these processes and thereby would have been specialized in any one of these **particular** streams. But by focusing on what lies behind all these processes, be it in environment or energy or fermentation or mineral or electronic namely gas-solid non-catalytic reactions, we are able to apply the same principles to all or any one of these fields and that is what makes us unique which distinguishes us from other disciplines of engineering such as mechanical, electrical or aero **and and** so on.

Now, let us turn our attention to find out how to handle such situations or what is it that we need to look at these situations. So, before we go on to look at the kinetics of these processes and things like that, let us first try to visualize how things are happening here and things are different, for example, take this carbon particle.

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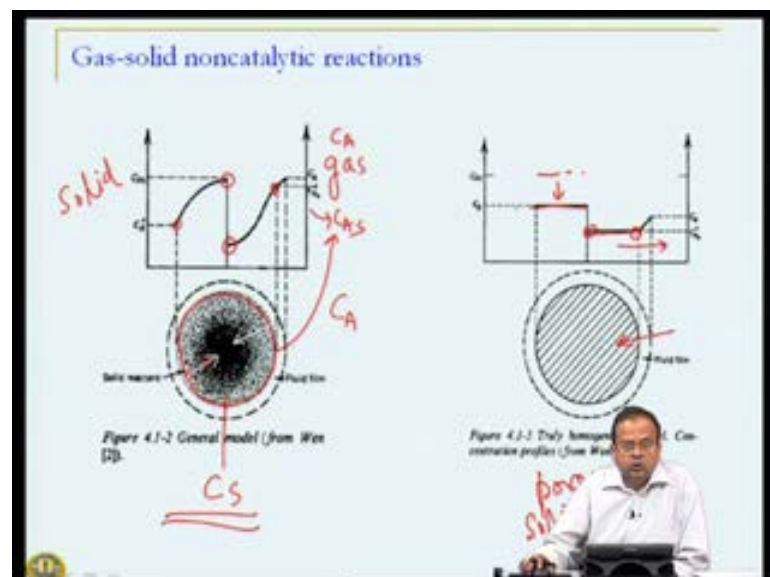
Let us say that we have a time t equal to 0, we have carbon particle and oxygen is burning it to carbon dioxide at some time t equal to one hour for example, we would have particle which is now reduced in shape.

If the initial particle size was, let us say capital R , at some point of time in future, its radius would be R which is function of time. Sometime later on, we will still have smaller particle and eventually nothing will be left. Entire carbon will be gone. That is an ideal situation, but sometimes, you have formation of soot and so on. That is an undesirable, but inevitable situation. The point I am trying to make here is that in gas-solid non-catalytic reaction, in these kinds of reactions, we have things changing with time, the size of this material or carbon particle, for example, changing with time. So, if you recall our catalytic reaction, particle was stable. In fact, all effort is done to make sure that particle does not change its size or chemical characteristic, where as that is not the case here. So, the concept of steady state which we often freely used in catalytic reaction can no longer be used over here. So, that is one type of scenario that we can think of.

Let us consider another example where we had, let us say a particle of Fe_2O_3 , we reduce it to get iron using carbon monoxide which gives us carbon dioxide. Now, what happens when this reaction is taking place? Suppose, this was that t equal to 0 at some point of time in future, we still have a same particle of the same size; however, depending on how the reactions are proceeding, we may have a core which is Fe_2O_3 , but a shell which is iron, because part of these Fe_2O_3 has got converted into iron. So, here particle size has not changed with time. Let us say that particle does not crumble, it stays as a particle, but the composition of the particle has changed.

For example, if it was pure Fe_2O_3 at the beginning of the reaction, at some point of time down the reaction, what would we have? We would have Fe and Fe_2O_3 . So, particle is now a mixed particle. How does it matter? If carbon monoxide was to diffuse in this particle, carbon monoxide and carbon dioxide coming out in same case over here, then, the diffusion characteristic of carbon monoxide and carbon dioxide coming and going out would be different at these different times. Why because, the porosity itself could be different. The iron may be more porous or non-porous than Fe_2O_3 . So, now, there is porosity which itself could be different. Of course, one could think of getting another scenario where the distinction between Fe and Fe_2O_3 is not sharp as shown here, but we have Fe and Fe_2O_3 spread all through the particle, everywhere, not just limited to a core like this. So, one has to be little imaginative in trying to figure out how we handle such situations or how we deal with such situations.

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In general, for example, let us see what can happen in a gas-solid non-catalytic reaction and what we see over here are schematics of what is likely to happen. So, what we see over here is some solid particle which is reacting and the gas is in the bulk. So, this is gas and this is solid and what we are seeing here is a concentration of the gaseous reactant on this particular axis and concentration of the solid reactant on this particular axis.

Let us say that we have gas. Firstly, we have a solid particle and let us say we consider the most generalized case. We have a fluid surrounding this solid and we will assume that all the resistance to mass transfer is located in this thin film which surrounds this solid particle. So, the concentration, let us say in the bulk is C_A , at the surface, the concentration will be little less. So, $C_{A,S}$.

This is concentration at the surface. Now, what happens as the reactant progresses through the solid? If the solid is completely or partially porous let us say, the reactant will diffuse. Now, as it diffuses, it will react with the solid, but starting with the outer shell of the particle. What will happen at the outer shell? At the outer shell, the solid concentration will be at its lowest value. Why? At the inner core, the solid concentration will be at its highest value because gas is diffusing in these directions and reacting and therefore, we will have. So, gases diffuse in this particular manner and therefore, we will have more reaction taking place at the outer periphery. So, lower solid concentration, but higher solid concentration inside.

If we look at the gas concentration, since gas is diffusing in this particular direction, we will have highest gas concentration at the outer surface and lowest inside. So, that is one kind of general scenario that is possible. So, what are some of the extremes of this?

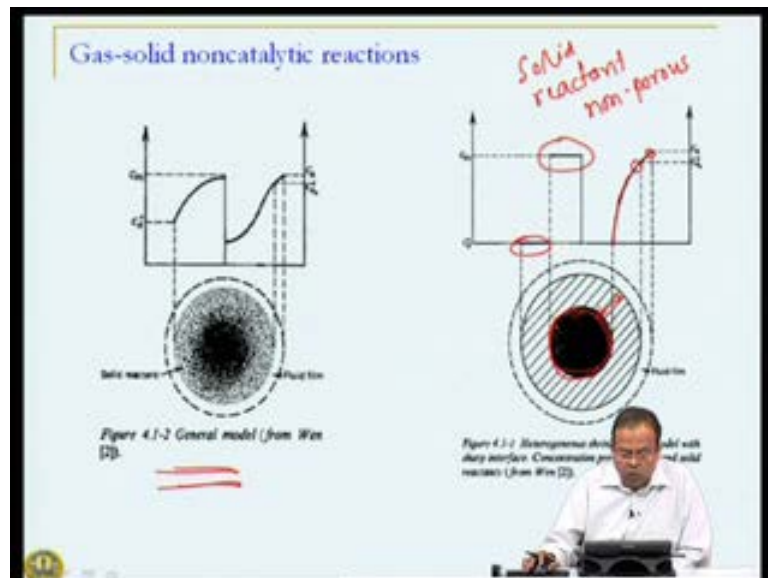
For example, let us consider a situation where this solid is extremely porous. Before we go further, I just want to point it out that once again we have the same feature that we saw in the case of gas-solid catalytic reaction namely, gaseous reactant diffusing and reacting. So, all those behaviors of effective diffusivity, tortuosity, porosity, all those concepts applied to this situation as well. The only difference is this solid is not static as was the case with catalytic reaction. This solid is dynamic in nature, that is, things are changing with time and that is the main difference.

Now, let us let us consider a situation in which, let us say that we have a porous solid. The solid is so porous that the gaseous diffusion is not at all controlled. Gas can freely

diffuse in and out and that is the situation shown over here. So, if gas **was** freely flowing inside, then the concentration of the gaseous reactant would be uniform, shown by this flat line, because, there is no diffusional limitation. It will be lower than the bulk because there is a reaction, but it will be uniform inside the particle. Now, what happens to solid? If the gaseous reactant concentration is uniform and solid initially was uniform, then the solid concentration also will be uniform throughout the **entire** core, something which is lower than the initial starting concentration, but uniform nonetheless.

Here, if this is the case, then this situation, there is no point recognizing that solid as a separate phase. Why because, diffusion is not limiting over here and therefore, we do not have solid **as** a separate phase that needs to be considered because it is uniform. So, its diffusion and other things are irrelevant as far as this situation is considered and when will this occur? This will occur if the solid is highly porous. So, that diffusion is **not** at all controlling **this** particular reaction. So, this is a case which we called as general, one extreme that solid is extremely porous, but what happens, to the other extreme if solid is non-porous?

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This is our general case, as before. But now, imagine a situation where the solid is completely non-porous and we are looking at a situation where we have non-**porous** solid. So, what will happen to the gaseous reactant?

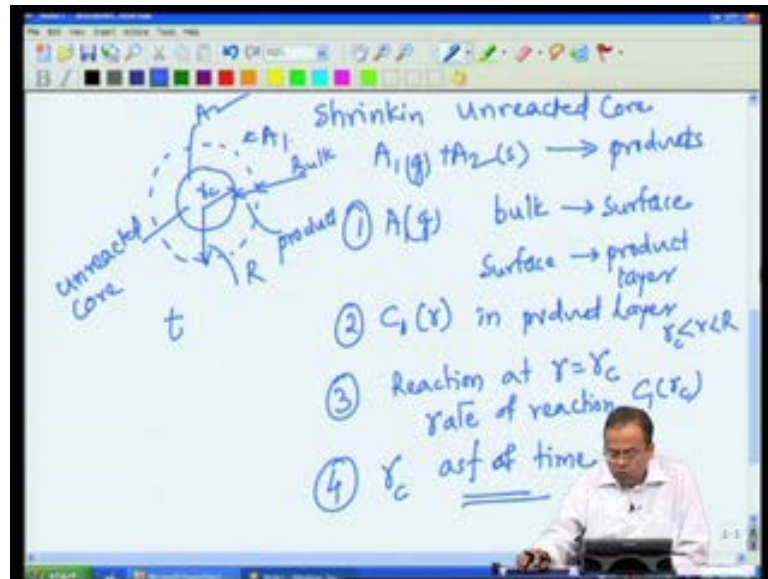
For example, we will come from the bulk to the surface, then, it will react with the outer periphery because it cannot diffuse inside. Now, when it reacts with the outer periphery, the solid from there will disappear and this **cannot** proceed further if that product that is formed is not porous. If product is also non-porous, then nothing can happen. It will just react at the surface and reaction will come to a halt.

If you want to know an example of this, you have seen silver articles becoming black, but when you polish it or you use some chemical treatment, you get back shiny silver material. What has happened here? What has actually happened here on the surface is the material is oxidized, fortunately this oxidation process cannot take place through and through solid material. If that was the case, then entire silver article from within also would be would be oxidized and blackened and therefore, the reaction is restricted fortunately only on to the surface. So, when you remove that surface either by polishing or by some chemical treatment, then, you have again shiny silver material inside which you can show. Of course, it is a different story that after a while, it may also get oxidized, but then, you have to keep shining it again and again. But unlike that, that is where the reaction actually comes to a halt.

But, in most general case, the product is likely to be porous. So, what will happen here is that the gaseous reactant will further diffuse into the solid. What will happen after sometime is, you have a product layer in which there is only diffusion. You have a surface on to which there is a reaction. So, the solid concentration in this entire zone is 0 because everything has got reacted. Solid concentration in the inside core is full solid because, reaction is only at the surface.

Hence, we have what we call heterogeneous shrinking core model. So, this is a solid core which is now shrinking in size. This will happen when the original solid reactant is non-porous. So, when we have porous solid, we have a general situation like this, when we have non-porous solid, we have a situation which is **something** of this kind

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So, let us now try to work out how this reaction actually proceeds. What are some of the key issues that we have to focus on? To do that, let us focus on this shrinking core model and this is also sometimes referred to as shrinking un-reacted core model. So, let us try to briefly look at how we handle with this kind of process. So, what we are going to see now is, how do we analyze this kinetically. It is something like this:

Let us say that at time t equal to 0, we had a solid particle, but at some time in future, we had a core. So, let us say that this is original solid of radius capital R , but at sometime t in future, only radius whose size core r_c is remaining. So, this is now a situation for which we need a kinetic analysis. So, this is shrinking un-reacted core model.

Let us look at what is the phenomenon or processes that are occurring. So, we have let us say, reaction A_1 going to A_2 , that is, reaction should also have solid. So, A_1 in the gas phase plus A_2 in the solid phase giving rise to some products. These products can be of different types, but let us just call them as product of some interest. Ok?

This is gas phase reactant which is coming from outside. This is A_2 which is reacting. What exactly is happening? We have A_1 . So, let us look at the first process. We have A_1 in the gas phase which diffuses from the bulk to the surface and from surface through the product layer. So, bulk product layer and this is un-reacted core. So, from bulk, it comes to the surface and then diffuses inside the product layer. We have concentration of the element A_1 as a function of R because, now, in the product layer, concentration will

be changing. We should know what this **concentration** is. Then, there is a reaction at the surface. So, reaction at R equal to r_c . **ok?**

We should know what the rate of reaction as a function of r_c is. But, is this r_c remaining steady? No, r_c is also changing with time. So, we have r_c as a function of time to be determined and this is where r_c as a function of time. This is where things differ **from regular** reaction. So, **what** all things we need to consider when we do this? Diffusion of gaseous component from the bulk to the surface, from the surface through the product layer which eventually will give us what the concentration is of C_1 as a function of R for r_c less than capital R . So, this is entire product zone. The reaction takes place at R equal to r_c . We need to know what C_1 of r_c is and from that, the rate of reaction and then, how r_c itself changes with time so that, we now complete this cycle. What I will do is, I will just give you a brief idea or just basic equations without going through the details of mathematics over here so that we see the bigger picture and not get lost in the details. We have for example, from surface within the product layer, there is diffusion. So, we can write as:

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$r=R \quad D_{1c} \frac{dc_1}{dr} = k_g (C_{1b} - C_1)$
 $r=r_c \quad D_{1c} \frac{dc_1}{dr} = -r_c = K C_1$
 $C_1(r) \quad r_c \leq r \leq R$
 $r|_{r_c} = K C_1(r_c)$ (3)
 solid $-\frac{dN_2}{dt} = r_c \cdot 4\pi r_c^2$
 moles $N_2 = \rho_s \cdot \frac{4}{3} \pi r_c^3$ (4)

For example, the **diffusion** equation for a spherical particle which takes of the form the steady state, let us say that we assume the diffusivity to be 0. So, this is the diffusion and there is no reaction in the product layer. So, we have this **particular** equation. What are the boundary conditions for this? Let me show the particle once again. What we are

concentrating on is the solid un-reacted core and a product layer and we are looking at diffusion in this product layer. So, this is diffusion equation. But, what is happening at outer surface? At outer surface, the flux of diffusion at this point must be counter balanced by the rate at which material is coming to the surface. So, this is external mass transfer coefficient K_g and we have $K_g (C_1 - C_1)$.

This is the flux balancing at the outer periphery. What is happening at $R = r_c$, that is, this particular point. Once again, the flux at that particular point now must be balanced by the reaction because there is no accumulation on the surface. It does not have any capacity to hold mass. So, no accumulation. So, flux at which the material is coming to the surface is the same rate at which it is reacting. So, what do we have here, this must be same as the rate of the reaction and let us say that rate of that reaction is a first order reaction, so, $K_1 C_1$. That is the rate that we have, but all this at $R = r_c$.

We would therefore, now require the solution of this equation to get C_1 as a function of R , for R between r_c and the outer periphery. This is fairly straight forward diffusion equation and we then need to know what the rate of reaction is. So, rate of reaction, this is as far as the first two steps are concerned. So, we have addressed these two issues. We need to know what the rate of reaction is at $R = r_c$. What is R ? We want to evaluate rate of reaction at r_c which is $K_1 C_1$ of $R = r_c$. Once we know C_1 as a function of this radius, we know what is happening to this r_c .

That is the core radius that is changing with time. We need a balance on the solid mass to see at what rate that radius of the core is changing. Why is the radius of the core changing? It is because of the reaction that is taking place on the surface. Suppose, we say that n is the moles of solid or mass of solid, then the reason why there is a loss dn/dt is because there is a reaction. So, reaction rate as a function of time and since reaction rates for gas-solid catalytic reactions are expressed in terms of per unit surface area, we have to multiply it by the surface area of the catalyst. So, this is the rate at which the solid is changing.

How do we connect this to get the equation for r_c ? What is this moles of solid? If we assume that the molar density is constant ρ_s , then, mass of solid is nothing but this density that is constant, let us assume that, into the volume of the solid and volume of the solid is nothing but $\frac{4}{3} \pi r_c^3$. So, that gives us the moles of solid. These

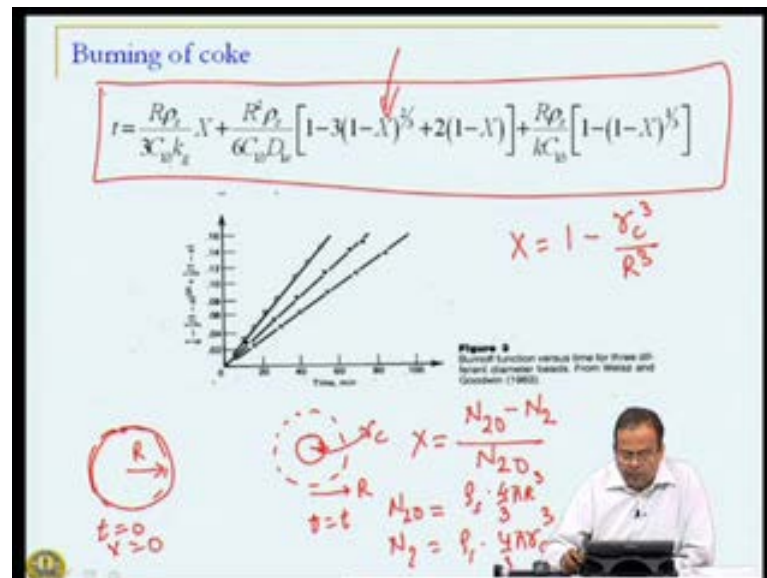
moles of solid put back into this equation will now tell us what will be the rate of change of this radius. So, we have $\frac{d}{dt}$ of $\frac{4}{3}\pi r^3 \rho_s$ which is nothing but $4\pi r^2 \rho_s \frac{dr}{dt}$ which will be nothing but rate at which reaction is taking place at the surface. This gives the desired equation for how the radius of the solid is changing.

This is my step four. This was step three. This whole thing was intended for step 1 and 2. What are these 1 and 2, three and four? Material or gaseous reactant moving from the bulk to the surface, from the surface within the product layer and then reaction at the surface and then R of C as a function of time. So, these are four steps which we have captured by writing the diffusion equation for the product layer combining it at the boundary at the external surface, that is flux are equal, internal and external and at the internal boundary, that is the core boundary r_c , the flux must be the rate of reaction. What is this rate of reaction? This rate of reaction is $k_1 C_1$ of r_c . Let us say that it is a first order reaction in gaseous reactant concentration.

We have this rate and what is a result of reaction the solid is losing its mass and rate at which solid is losing its mass must be the rate of the reaction and expressing the solid in terms of its molar density into the volume, we get an equation of how r_c changes with time.

When r_c changes with time, what can we do further? We can now say that at time t equal to 0, r_c is equal to capital R and at any time t R is equal to r_c . We have a single equation which we can integrate between these two boundary conditions and the equation is little messy, but let us try to look at the final form of this equation before we look at this particular example.

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So, what we see here is that if you integrate that particular equation, this is what we will get. I will just explain what **this** is. This is that equation. There are few terms that you see here which probably are not familiar at this point, but, let me explain what this is. What we are saying in this particular gas-solid catalytic reaction is:

What is it that I am interested to know, for example, if it is a burning of coke or if it is a burning or smelting of nickel sulphide, what is the **conversion** of coke, coal or nickel sulphide or Fe_2O_3 and so on. How do we define conversion here? As I said, this is a time dependent process and for time dependent processes such as batch reactors, you always try to relate conversion with time. That is, given time what will be the conversion or to achieve certain conversion, what is the time that is required? How do we define conversion for a gas-solid non-catalytic reaction?

For example, the previous case which we are seeing, suppose this was particle of radius R to begin with, at time t equal to 0. At time t equal to t , what I was left to it was the product layer within which there is a core. So, this product layer still radius R , but this is r_c . So, how do I now **define** conversion? We can define conversion in the same way as we define for any species. What was that, if into 0 was the moles of solid to start with, minus into is the moles that are remaining divided by into 0.

That is my conversion x and that is that x that you see over here. Now, how do we get that x in all those **crazy** forms over there? That is because, initially, let us say the moles

were again going back to solid definition ρ_s is four by three pi R cube. So, this is N_2 and what is N_2 ? N_2 will be ρ_s into four by three pi r c cubed. If you put these two values, then my conversion here will be 1 minus r c cube by R cube. That makes sense. At the start of the reaction, r c is equal to R. So, conversion is 0 at time t equal to 0 and at sometime in the future, r c is what is remaining. So, 1 minus r c cube by R cube is the fractional conversion of the solid. When r c goes to 0, the entire solid has reacted.

Now, some of the terms that you have seen before C 1 b for example, is a concentration of gaseous reactant in the bulk, K_g is the mass transfer coefficient, ρ_s is the molar density, D_1 is the diffusivity and so on.

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moles solid

$$N_2 = \rho_s \cdot \frac{4\pi r_c^3}{3}$$

$$-\frac{d}{dt} \left(\rho_s \cdot \frac{4\pi r_c^3}{3} \right) = -\rho_s \cdot 4\pi r_c^2 \frac{dr_c}{dt}$$

$$= \gamma(r_c) 4\pi r_c^2$$

$\Rightarrow t=0 \quad r_c=R \quad t=t \quad r_c=r_c$

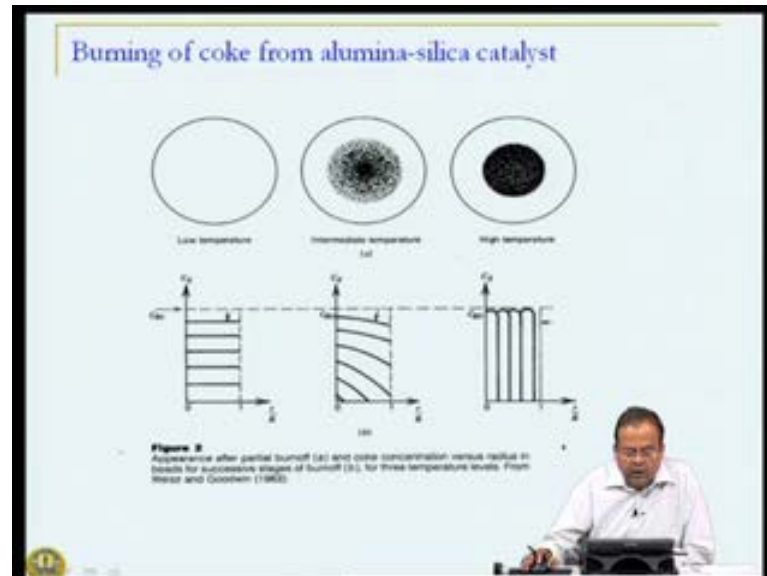
$$t = f(r_c, R, D_1, k_g, K, \rho_s)$$

What happens is, if we solve this equation, you see all those terms over here, D_1 , K_g , r_c , ρ_s and so on. When we integrate this equation with this boundary condition, one can actually get t as a function of r c, capital R and all the properties that are involved over here, the reaction rate constant, ρ_s and so on. This functional form is what you see over here and it gets nicely arranged in the following manner.

Let us start with this last term. What is this last term representative of? If you look at this last term, that has the reaction rate constant. This is a contribution from reaction. As you can see over here, the second term has diffusivity in it and so, we can clearly see that must be contribution due to internal diffusion and first term is a contribution due to external diffusion. So, intuitively, it makes sense because, the process has external

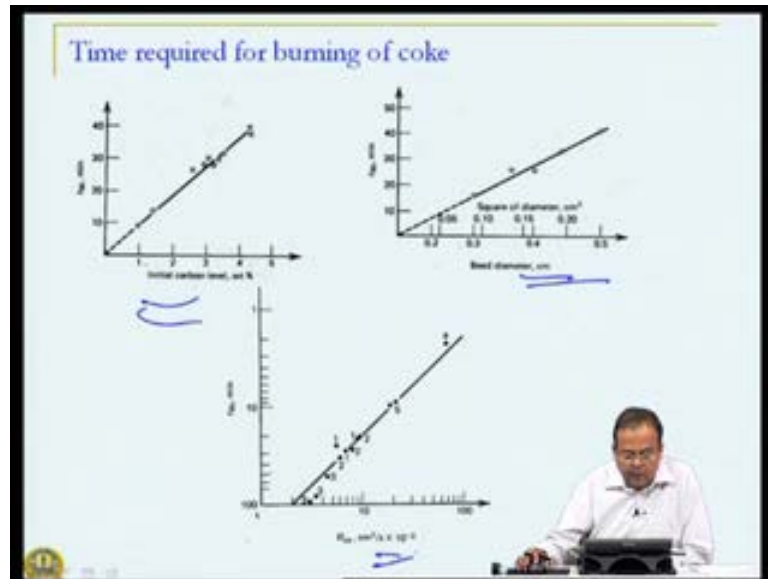
diffusion followed by internal diffusion reaction. So, the time that you see here is a contribution from all these factors in an additive manner, that is, resistances in series kind of situation. We had seen this kind of pattern even before.

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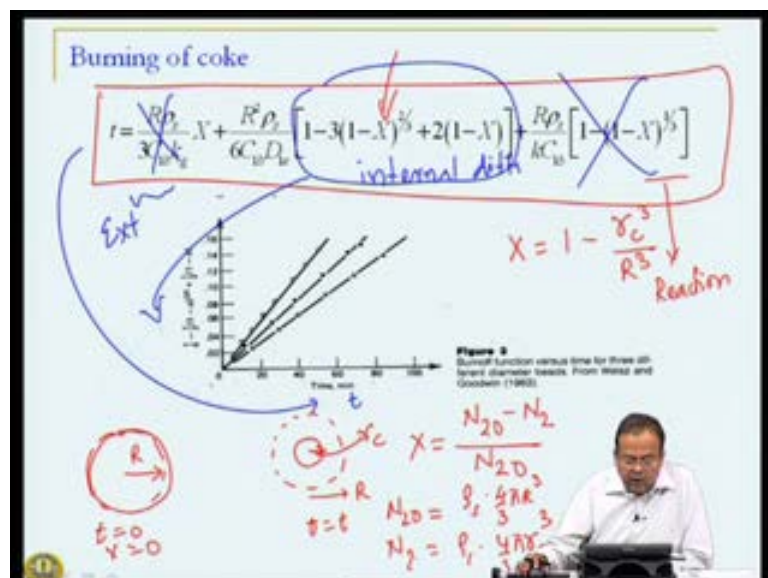
Now, how do I make use of this? This is an example of burning of coke from alumina-silica catalyst. Catalyst has some carbon deposited on it. Here, you are not trying to generate energy, but you are trying to regenerate catalyst. So, catalyst has coke deposited on it and you are trying to burn it off. So, **what** you can do is, you can plot time verses conversion. Now it turns out that, you cannot do conversion for hundred percent. So, you can look at eighty percent conversion..

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Now, if I plot in this particular case, this parameter which is on the y-axis verses time, which is on the x-axis for **different** sizes of the catalyst particle, you see that there is a straight line; that means, in this particular case, internal diffusion is controlling because contributions due to these two terms is **negligibly** small and similar experiments for example, **were** done **with** changing the initial **carbon** level changing the bit diameter, changing the effective diffusivity and so on.

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In each case, you get a straight line. Once again, all those things are coming from here. Carbon content will change the ρ , S , diffusivity is changing over here, bulk concentration is changing over here, radius of the particle is changing over here. So, all those factors you still get a **similar** straight line indicating that internal diffusion is controlling in this particular case.

We will stop here for this particular session and look at what are two more cases of gas-solid reactions in the next session. So, let us recap what we saw in today's session. We saw lot of examples where gas-solid non-catalytic reactions are important. Few examples involved even liquid, but we will come to liquid little later. You also saw that gas-solid non-catalytic reactions share common features with gas-solid catalytic reactions namely diffusion both external, internal diffusion reaction and so on, But, there is a major difference between non-catalytic and catalytic reactions and that has to do with what happens to the solid. In a catalytic reaction, solid was static. It did not change and therefore, we could easily assume for steady state process, but in non-catalytic reaction, that is not the situation. In non-catalytic reactions, solid is continuously changing. It could be an extreme case of solid disappearing all together or if not, the composition of the solid keeps changing.

So, this is what makes gas-solid non-catalytic reaction a dynamic process or a time dependent process and therefore, we would focus on how long does it takes for particle to react. If you are looking at burning of coal for example, how long does it take for this coal to burn is one of the major questions that need to be answered while dealing with gas-solid non-catalytic reactions. The way to answer that question comes from the rigorous analysis of this process and in doing so, there are several scenarios possible. Let us say that we have a solid of fairly good porosity or moderate porosity, then, what will happen is the gaseous reactant will diffuse inside the porous solid and it will react differentially within this porous solid because, the concentration of the gas-gaseous component will be different along the radius of the solid let us say.

So, that is a **general** scenario, the extreme case of that is when solid is so porous that diffusion does not play any important role at all, in which case, the entire solid is a homogeneous mass and we can treat it just as we treated any homogeneous system. We do not have to consider any solid presence separately over here. The other extreme is where solid is so non-porous that we have completely no diffusion of reactant taking

place inside the solid. So, reaction is confined to the surface of the solid, but as time progresses, **as** the reaction progresses, the core or the surface at which this reaction takes place will shrink because the solid is getting reacted and therefore, we have a situation of shrinking un-reacted core model which we saw in the last example and we analyze that to see how time is related to the conversion.

In the next session, we will look at yet another situation where we have, what we call shrinking reacted core model or situation and we will try to analyze this. **Thank you.**