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# Module No. # 10 Lecture No. # 26 Chemical and Compositional Characterization: Reactivity

Welcome to the twenty-sixth lecture in the particle characterization course. In previous modules in this course, we have looked at various aspects of particle characterization starting with morphological characterization. Then, we looked at structural characterization, interfacial characterization, transport property characterization; today, we are going to start discussing chemical and compositional characterization of particles.

Particles are widely used in industry for the purpose of either participating in chemical reactions or promoting chemical reactions in the form of a cap list. Now, when particles are used as reactants, the primary reason for that, is the chemical reactivity of a material increases as you reduce its size, and as you take the same amount of material for quantity or volume of material, and provide it to the reaction in finer and finer particular form, the overall efficiency that you achieve per unit of particular material, increases significantly. And that is the reason why it is so attractive to use materials in powder form or particulate form in most chemical engineering processes.

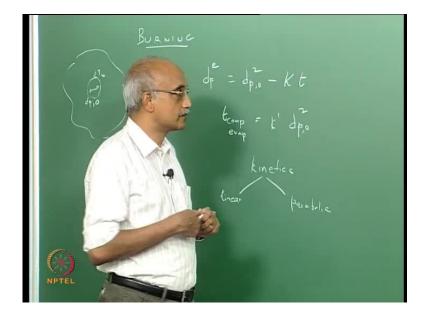
Now, when we look at chemical processes that involve particles, there are predominantly three modes in which they participate in chemical processes. The first is burning solid particles as well as liquid droplets that are exposed to a hot fluid; a hot gas will evaporate particularly in combustion where you want the fuel to burn as quickly as possible and also with as little supply of a oxidizer as possible. Droplet combustion is considered to be much more efficient and effective compared to trying to burn sheets of liquid. So, combustion is an example of where particle burning is widely resorted to in order to achieve high combustion efficiencies.

The second type of process involved in particle... is dissolution - when a solid particle is immersed in a liquid, it tends to dissolve in the liquid. Now, this is not just an industrial process but, it is something we use in our household every day. When we take sugar and

dissolve it in milk or you take coffee powder and you dissolve it, they are all examples of dissolution processes involving particles.

The third type is sublimation. Sublimation is where a particle will directly be converted to its vapor form without going through the intermediate liquid phase. So, of the processes that involve chemical change of a particle, the 3 most widely used are burning resolution and sublimations. We will take a look at this process in some more detail in this lecture and we look at burning of droplets.

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We consider a situation where you have a droplet or a particle of some initial diameter d p 0 which is surrounded by a hot gas let us say, of temperature T infinity and the temperature of the droplet is some t w. The droplet which is at a lower temperature will begin to evaporate as soon as it experiences the higher temperature of the surrounding gas and similarly, it is a particle - solid particle. It will start burning; also, now you may be familiar with the expression for the burning rate of a particle: d p squared at any time t equals d p 0 squared minus some k t; where k is a constant and t is time. Now, from this you can also extract t for complete evaporation, by simply equating d p to 0 and when you do that we simply get some k prime of d p 0 square. In other words, the time required for complete combustion of a droplet or a particle drops as a size of particle or droplet, decreases with a square dependence. So, there is a huge benefit to be had that trying to speed up a combustion process or an evaporation process or burning process

that is a huge leverage in the size of the droplet. The finer droplet size or finer the solid particle size, the faster it burns.

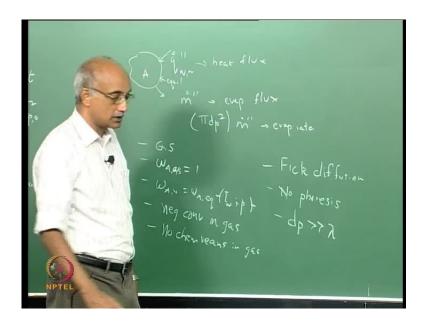
Now, this expression is something that we are all familiar with but we need to spend a little bit of little bit of time understanding how we arrive with this expression. I mean, what is the underline mechanism that causes this type of relationship to happen. When a particle especially is burning there are essentially 2 models of particle burning. The first is a linear model and the other is a parabolic model so, the kinetics of burning of droplets and particles can be classified as linear and parabolic.

The difference between the 2 models is, in the first case you assume that there is an infinite supply of oxidizer that is constantly available to the droplet, so that the rate at which the droplet is burning is only limited by the exposed surface area of the droplet. Whereas, in the latter model the parabolic model the products of burning or combustion essentially start forming a coating on the outside of the particle which means that now the combustion products have to be diffused out and oxidizer has to diffuse in.

So, the process of burning or the rate of burning becomes limited by growth or build-up of this product layer around the external circumference of the of the particle or droplet that is burning. So, this introduces essentially, a self-limiting factor and these 2 models are again widely used to consider the 2 different cases where you have an excess of oxidizer versus an excess of fuel. So, the linear model is typically applied when you have an excess of an oxidizer and the parabolic model is the applied when you have fuel rich condition.

Particularly the parabolic model is applicable when you are burning solid fuels whereas, the linear model is typically to use when you are burning liquid droplets. Especially, as the droplets get finer and finer you can essentially assume that the process itself is not going to be limited by the buildup of a product layer around the circumference.

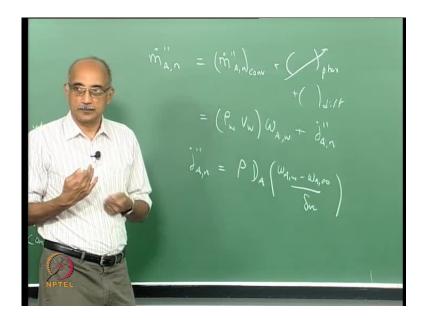
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But, when you look at this problem of particle burning or droplet burning, it is essentially a process that is initiated by diffusion of energy from the hot gases. So, the energy transfer from the hot gas essentially provides the latent heat of vaporization that is required to convert the liquid droplet or the solid particle into its vapor form. The transport phenomenon that is involved essentially is, you have heat of some flux q w dot double prime. Let us just look at the normal direction readily outvert and invert direction so this is your heat flux that is being supplied (Refer Slide Time: 09:00) to the droplet or the particle and this causes a burning of the droplet or particle which is represented by an m dot double prime. Again, following of the notation we have used in this course the dot stands for unit time and double prime stands for the unit area. The heat flux that is supplied by the hot gas to the droplet causes a mass flux in the in the form of evaporation from the droplet or particle into the hot gas and if you take this m dot double prime and you multiplied by the diameter of the particle or droplet, that gives you the instantaneous evaporation rate at any time. Now, clearly then these 2 are related; the amount of heat that you are providing determines rate at which evaporation is happening and therefore, any expression that you write for evaporation rate as a function of a time must include the component of heat flux has been supplied to the droplet.

Now, if you look at the overall conservation equations for this problem and you look at the mass conservation equation, let us make some assumption. Let us assume that we are looking at a quasi-study state. So, we do not assume that the processes of heat supply and a droplet evaporation or in dynamic balance and let us assume that this liquid, solid droplet or a particle, is a pure substance; let us call it some substance a. so, omega a in the liquid phase or solid phase is equal to 1; that is the mass fraction of substance a in the solid or liquid phase is equal to 1; let us assume that equilibrium prevails at the interface between the droplet and the surrounding gas. Omega A W is equal to omega; omega A at equilibrium at heat prevailing temperature and pressure. Let us assume that the surrounding gas has negligible convection that is, both natural convection as well as forced convection or relatively low in magnitude. Therefore, the combustion process or the burning process is essentially controlled by diffuse a phenomena. It is also assumed that there are no chemical reactions in the gas phase. So, once the material a leaves the solid particle or liquid droplet and enters it into the gas phase, there are no further chemical reactions that convert a to b or c or so on and let us assume that the diffusion process itself is a simple diffusion process. In other words, we are not considering any phoretic effects; we are not considering a thermal diffusion for example.

So, under and the other assumption is of continuum. Let us assume that particle diameter is much smaller than or much larger than the prevailing mean free path in the gas. So, even though the droplet itself may be very fine it is still very large on the scale of the mean free path of the surrounding gas.

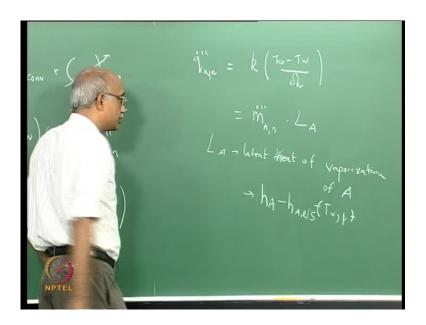


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When you make all these assumptions, let us look at the expression for this evaporative flux of the droplet. Now, let us write it as m A n dot double prime which is the flux of the pure substance a in the radially output direction. This then if you recall the expression we wrote in the last class, will have 3 components. There is a convection component, there is a phoretic component and there is a diffusive component. So, you have a m dot A n convective plus same phoretic plus the same in diffusive mode.

Now we have assumed that for a phoresis is absent; so that term drops out and the expression for the convective transport of species A is the convective transport of the vapor that is generated. Let us call that some rho w V w, where V w is the radially outward velocity of the vapor that is generated due to the evaporation process, rho is the density times omega A w; that is the mass fraction of a in the vapor phase at w. so, this is your convective portion plus the diffusive portion. Now, the diffusive portion is let us write it as some j A can dot double prime, where this diffusive term will be equal to row times D A times.

Let us make one more assumption that your thermo physical properties are constant across the diffusion path. So, that normally we would write a gradient in mass fraction for the Fick diffusion expression; if you make this assumption you can essentially replace the differential gradient by a difference expression. You can write this as omega A w minus omega A infinity over A m - A mass fraction or a mass transfer boundary layer thickness. Here, rho is the density, D A is the Brownian diffusivity of species a in the hot gas, omega A w minus omega A infinity is the difference in mass fraction of species a from the outside of the droplet w to far away from the infinity. So, it is that gradient in mass fraction that is driving the process of evaporation. (Refer Slide Time: 16:53)

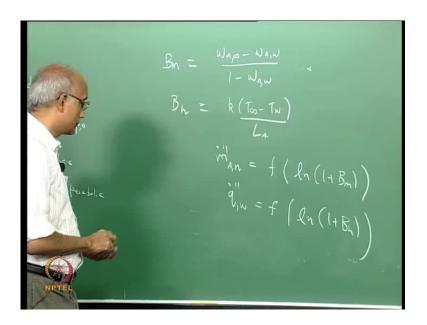


The overall expression then for the evaporation flux of species a is rho w V w times omega A w plus rho times D A times omega A w minus omega infinity over delta m.

Now, correspondingly that is a heat flux that is supplying the energy to drive the evaporation process and in the case where its simply a Fourier heat flux, we can write this as k times T infinity minus T w divided by a heat transfer bound layer thickness. Let us write that as some delta h. So, what is the relationship between this and this (Refer Slide Time: 17:12)? Well, this heat flux must then be equal to m A dot double prime in the normal direction times L A where L A is the latent heat of vaporization of substance A; because the latent heat of vaporization itself is the difference in the enthalpies of h A. That is, in the vapor phase minus h A in the solid or liquid phase at T w and a pressure. So, it is actually the difference in enthalpy for the species a being in vapor phase minus the species a being in liquid or solid phase. That is the definition of latent heat of vaporization.

So, this then provides the link between the rate of vaporization of the droplet and the flux of heat that is being provided to the droplet to promote its burning. Based on this you actually write an expression for the evaporative flux of material a in terms of essentially the latent heat of vaporization - the temperature differential between the droplet and combustion or the hot gas which is actually driving the evaporative process.

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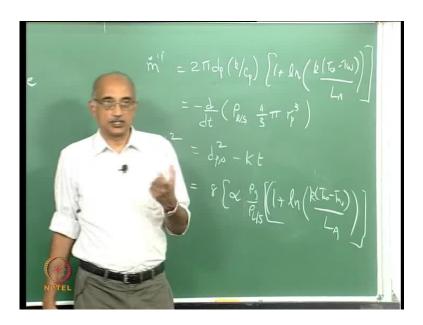
This can also be represented in terms of certain dimensionless parameters that is, a parameter B m which is called the blowing parameter; or mass transfer which is written as omega A infinity minus omega A w whole over 1 minus omega A w. Correspondingly, there is a blowing parameter for heat transfer which is written in terms of k times T infinity minus T w over the latent heat of vaporization L A and this parameter essentially, controls the diffusive flux. And in fact, the m dot double prime A n can be written as a function of l n of 1 plus B m.

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 $e = \frac{1}{2}$   $S_{c} = \frac{1}{2}$   $P_{r} = \frac{1}{2}$ 

Similarly, q dot double prime can be written as a function of 1 n of one plus B h. It is turns out that B m and B h are also related to each other; 1 plus Ln of 1 plus B h can be written as 1 n of 1 plus B m to the power Louis number, where Louis number is equal to Schmitt number as is equal to nu by d and Prandtl number is equal to nu by alpha. Schmitt number is where nu is the kinematic viscosity, D is the diffusivity or the Fick diffusivity and alpha is the thermal diffusivity; the Louis number is actually D by alpha; relating Fick diffusivity to thermal diffusivity. So, when you take these expressions together, you can finally extract a relationship between the evaporative flux and the heat transfer parameters which I have mentioned earlier, are easier to measure.

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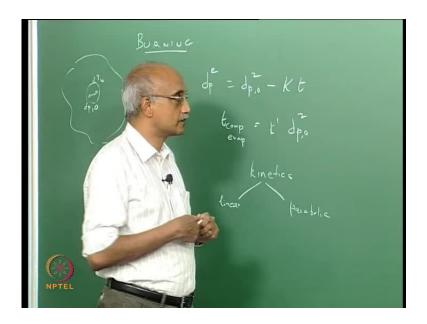


So, m dot double prime then can be written as 2 pi d p k by c p times 1 plus logarithm of k times T infinity minus T w over L A. This is the expression that directly relates the evaporative mass flux to the particle size k by c p, where k is the thermal conductivity and c p is heat capacity at constant pressure and latent heat of vaporization 1 a as well as the temperature differential T infinity and T w. Now, this is the evaporative flux but, then this is what is then driving the rate at which the mass of the droplet is changing. This is equal to minus d by dt of what is the mass of the particle. It is a rho 1 or rho solid whatever the, whether the material is the liquid or a solid times its volume. Now, what is the volume of the material? It is basically 4 by 3 pi times r p cubed.

This then is in terms of this is mass divided by time. This actually, if you calculate this equation, this also, a mass transfer rate; so, it is evaporative. The rate m dot prime which now must be equated to minus d by d t of rho times 4 by 3 pi r p cubed; when you do that and now you integrate for r p or d p, that is when you extract the expression that we had written down earlier. The d p squared is given by d p 0 squared minus k t, where the k value now can be extracted from solving this equation as: 8 times alpha row g by row 1 times 1 plus logarithm of k times T infinity minus T w over L A.

Basically, what we have done is, comparing to the original equation we have now derived an explicit form for what that k is and again, the key thing that you should remember, not necessarily the equation per say, that dependences the terms that are involved. alpha is a thermal diffusivity which is equal to k by row c p row g by row l is the ratio of the densities of the gas and the liquid or the droplet, I mean, or solid and you have latent heat of the vaporization of the material in the denominator; k times the temperature differential T infinity minus T w in the numerator.

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So, this is the fundamental basis for how this equation is arrived at but, in its simplest form, this can be obviously obtained. Experimentally also, all you have to do is, measure droplet diameter or solid particle diameter as a function of time and you can extract what this constant is. The Burniuc constant is a key parameter in determining the combustion efficiency of any droplet or particle. Now, finely divided particles tend to burn very fast; which solid materials burns fastest.

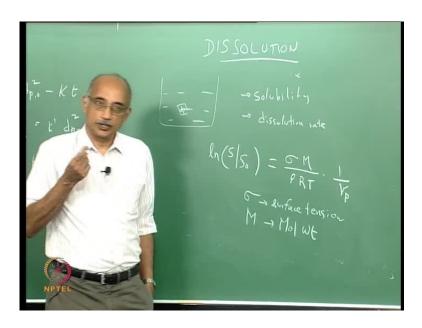
Magnesium tends to auto ignite; so there are few solid materials that are also easy to ignite and they have very high calorific content. Now, the reason that magnesium is so easy to ignite has to do with its latent heat of vaporization. It can burn even with the supplied relatively little heat compared to something like aluminum or iron which will take a lot of heat energy to start burning. On the other hand, liquids typically burn much faster and especially high vapor pressure hydrocarbons; therefore, are attractive as fuels because with very little supply of heat from the hot gas and with very little supply of an oxidizer, they will burn virtually spontaneously.

On the other hand, in order to get a water droplet to vaporize, actually it takes a lot of energy input compared to getting a hydrocarbon fuel to vaporize. Actually, the most combustible fuels turn out to be cryogenic liquids like essentially, these are liquids that as soon as you expose them to atmospheric conditions, or near room temperature they will burn with the very high evaporative losses which is good from a combustion efficiency view point. So, for example, for satellite propulsion, cryogenic fuels are used widely compared to other types of fuels like kerosene and so on.

In terms of combustibility it is a physical process in one sense but the chemistry really enters through this term. The latent heat of vaporization that is really what distinguishes the rate at which different droplets and different solids burn relative to each other. So, we have talked so far about burning which is one type of transformation of a solid or liquid particle into gaseous form. Now, the other frequently encountered situation is dissolution - where you have particularly a solid particle material that is immersed in a liquid and you are interested in the rate at which it will dissolve into the liquid. Frequently you want to maximize the rate; you want the particle to dissolve as quickly and as completely as possible in the liquid.

Now there are really 2 terms that affect the dissolution process. The first is solubility and the second is rate of dissolution.

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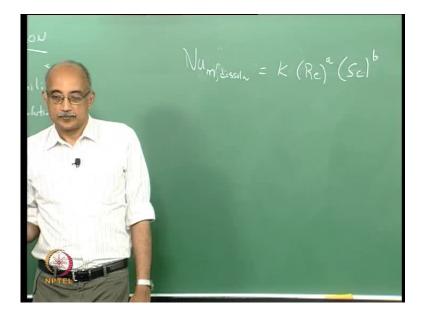


So, let us look at particularly how the size of the particle affects these 2 parameters. So, this is the case where you have a liquid in which a particle is suspended and the particle is dissolving into the liquid. The 2 terms are solubility and dissolution rate; so the solubility establishes an equilibrium limit as to what is the maximum amount of material that can dissolve into the liquid. Whereas, the dissolution rate provides a kinetics of the dissolution process; turns out, the both of them are related to the size of the particle once again. The expression for solubility in terms of particle size is written as 1 n of S over S 0 equals sigma M over rho R T multiplied by 1 over r P, where sigma is your surface tension parameter, M is the molecular weight of the solid, rho is the density of the solid, t is the temperature and r is the radius of the particle; s in the denominator is the actual prevailing solubility, S 0 is the solubility in the case of very large chunk of the material.

Theoretically, infinite dimensional material and the solubility corresponding to that so basically, what we get from this equation is, the solubility is inversely related to particle size so the finer that you make the particle the greater is the solubility of the material into the liquid and you can see that there is also the surface tension this relates to the ratio between solubility of a particle to the solubility of the continuous layer or continuous substance.

So, clearly the largest differentiator in this is the particle size; now, so this suggests that you should use fine particles if you want to improve the dissolution characteristics but,

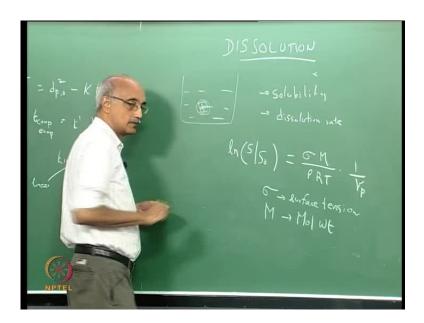
only from a maximum dissolution view point the rate of dissolution is governed by as we saw in the last couple of lectures and Nusselt number.



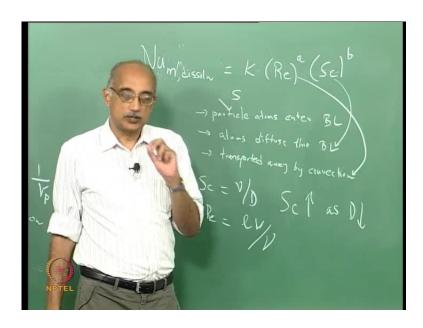
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So, the Nusselt number for mass transfer for a dissolution process can be written as some constant times the Reynolds number to the power some a times Schmitt number to the power b. Again, we saw in the last lecture the Nusselt number which is dimensionless expression of mass transfer in the case where it is dominated by diffusion; is related to two other non-dimensional parameters - the Reynolds number and the Schmitt number. Now, in this expression if you look at it carefully the dissolution process itself can be looked at as consisting of 3 stages. In the first stage, the particles from the atoms and molecules from the particle must first detach themselves from the particle and enter the liquid and also there is a boundary layer that will surround this droplet or this particle that is immersed in the liquid. So, the first step is that particles or particle atoms enter the boundary layer that is surrounding the particle.

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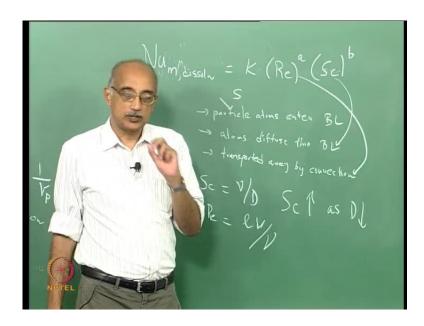


The second step is that these atoms or molecules diffused through the boundary layer to its outer edge and then, once they reach the outer edge further transport of these atoms away from the solid can occur by convection. So, if we look at this expression for the Nusselt number that governs the overall resolution process, the Reynolds term represents the effect of convection. Whereas, the Schmitt number affects the rate at which the atoms diffused through the boundary layer and what is the term that controls the first step here. Particles entering the boundary layer that is, the solubility term; that is why S is important. So, the higher the solubility, the higher is the Nusselt number; the higher the Reynolds number; the higher will be the Nusselt number and higher the Schmitt number, the greater will be the Reynolds number. Now, the expression for Schmitt number is nu by D; so for a given liquid in which the particle is dissolving, the lower the diffusivity of the particle, the greater will be the Schmitt number. So, the Nusselt number for mass transfer has an inverse relationship to diffusivity. So, Schmitt number will essentially increase as D decreases.

Reynolds number on the other hand, why does the Reynolds number enter the picture? The Reynolds number is involved when you actually setup a convective process to speed up the dissolution. For example, if you are trying to dissolve sugar cubes in coffee, what do you do you stir it? So, what does stirring do? Stirring, essentially increases your effective Reynolds number. So, this is a value that depends on 2 things; one is the stirring speed and what is the characteristic dimension? I am sure you have noticed that length of the stirrer makes a difference or some physical dimension of the stirrer also makes difference to the rate at which dissolution happens. So, 1 v over nu where I and v are both related to the stirring process itself. The length of the stirrer, the characteristic dimension of the stirrer, a characteristic velocity of stirring or in the numerator and the kinematic viscosity of the fluid is in the denominator.

So, the Reynolds number then, is an additional enhancement. I mean, dissolution will happen even if there is no stirring but, it will be much slower and temperature and the size of the particle will make a big difference. Again, if you have very fine sugar for example, granulated sugar - it tends to dissolve much more easily than if you have large chunks of sugar in your liquid and similarly, if you heat up the liquid then the dissolution process is much faster and so, dissolution itself is a process that is controlled by all 3 steps: the diffusion step well the solubility step is first one if you have low solubility it does not matter.

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You know how much you stir and so, you have to have a solubility material. for example, if you have a completely insoluble material in the one extreme case

It is never going to dissolve solubility is a key parameter the particle has to have a thermo dynamic

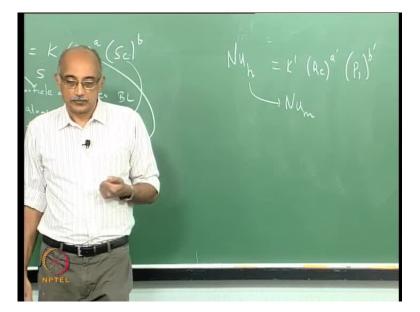
Feasibility of dissolving in the liquid so the solubility essentially reflects what is the equilibrium concentration of particle that can enter the liquid through the dissolution process these 2 are the rate limiting steps in the case where you have essentially a quiescent liquid in which the particle is dissolving the diffusion process becomes the critical process you in order to get a high rate of dissolution you have to maximize the rate at which diffusion happens in the liquid whereas, if you have the ability to add a convective force to it then, you can significantly speed up the process.

Now the Reynolds number increase can be achieved not only by stirring but also by other means of providing agitation. For example, you can do something like a vibration; so high frequency acoustic vibration is frequently used to again improve dissolution processes and the way that works is actually to-fro unlike stirring which really only affect the convective portion. If you actually add an acoustic process, a vibratory process it will have 2 affects. It will induce a convective effect but at the same time it will also reduce the thickness of the boundary layer min and therefore, acoustic methods can

enhance both the convective portion as well as the diffusive portion of the dissolution process.

So, if you look at industrial processes when you have for example, packed beds and fluidized beds in which your passing a liquid through a bed consisting of soluble particles and the rate of dissolution of particle into the flowing liquid is a critical parameter that has been well studied in literature. This relationship linking the Nusselt number to Reynolds number and Schmitt number, has been established particularly for conventional chemical engineering processes such as packed beds and fluidized beds. However, in the case of any process that involves dissolution of a solid into a liquid, the experimentation that is involved in this case is fairly straight forward.

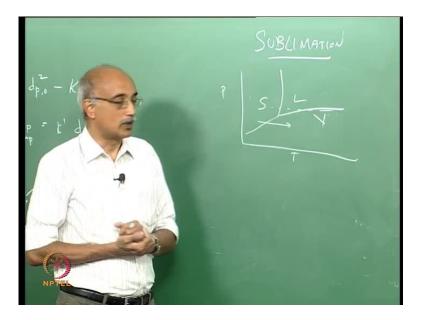
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However, again you might want to think about establishing an analogy with heat transfer and thereby extracting the Nusselt number. For example, if you want to estimate the Nusselt number governing dissolution and you want to do it experimentally, it may be easier to for example, look at the heat flux and derive an expression for Nusselt number for heat flux has some constant times Reynolds number to the power a prime Prandtl number to the power b prime, which can be done much more conveniently. I mean essentially, the way you do this is that you will take a particle, immerse it in a liquid and essentially look at how temperature equilibrates and this can be done even with insoluble particles. And, based on your calculation of the Nusselt number then as we saw earlier in the case where there are no phoretic phenomena and no inertial phenomena which is the case in the case of simple dissolution of a powder in a liquid. Once you have established the Nusselt number for heat transfer from this, you can extract the Nusselt number for mass transfer by assuming that the functional dependence is the same for both heat transfer as well as mass transfer.

Now, the analog of this is sublimation which is essentially the dissolution; so to speak of a solid in a gas where the solid turns directly into gas and typically that happens under thermodynamic conditions where you are below the triple point of the material.

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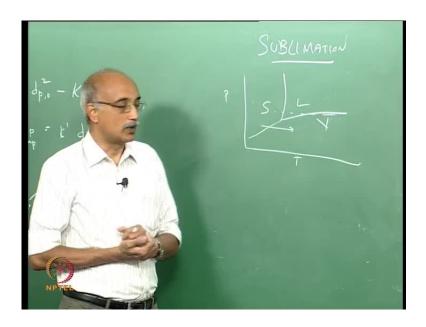


If you plot temperature versus pressure and you have some behavior like this where we have solid and solid, liquid and vapor, essentially, as you increase the temperature you should normally go through a transition from solid to liquid to vapor. If your pressure is very low you can directly enter from solid to vapor and that is basically called sublimation. Now, sublimation is again, if you think about it - a process that is very similar to dissolution in the sense that 3 things have to happen. The thermodynamics has to favor the spontaneous entry of atoms from the particle into the gas phase so there should be a tendency for spontaneous evaporation of the material into its solid form. And then, just like you have liquid boundary layer surrounding particle in the case of dissolution, you will have a gaseous boundary layers surrounding the particle in the case of sublimation.

Just like you can promote dissolution by introducing convection you can also promote sublimation by introducing a convective process. So, the discussion of the Nusselt number for dissolution and the Nusselt number for sublimation, are very similar and just like we talk about the solubility of a particle in a liquid we can also talk about essentially the viability or the feasibility of a solid particle or a solid atom spontaneously entering the vapor phase; so the thermodynamic constraint also applies to sublimation typically this step is the critical step in the sublimation because, unless the thermodynamic conditions are favorable I mean, it starts with the phase diagram. If you happen to be in a temperature pressure regime where sublimation is not possible, it does not matter if your transport conditions are designed to be favorable to sublimation.

So, unlike dissolution where as long as a particle is soluble in a liquid, it never becomes a rate limiting step and these will become the rate limiting steps. Sublimation is different in the sense that thermodynamically it requires a very specific set of conditions that need to be achieved in order for sublimation to be initiated. Once it is initiated, the rate at which sublimation proceeds will again be governed by a Nusselt number for mass transfer. Typically, both in the case of dissolution a well as sublimation, the Stanton number is not a critical factor because, you are considering typically cases where the flow velocities are relatively low and therefore the process of dissolution or sublimation is governed by diffusive phenomena. There are always exceptions of industrial processors where the Stanton number or the convective flow can dominate dissolution and sublimation; but, it is fairly unlikely. On the other hand, the process of fuel combustion it can be also governed by convective phenomena.

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In the simple model that we developed that established an essentially a quadratic relationship between particle size and burning time, the assumption was that convection was negligible but in that case also you can imagine a case where if there is a significant amount of convection that is present to aid in the burning of the droplet, the burning rate can be substantially enhanced because of the presence of that convection. This can be achieved either by directing the droplets at very high velocities, so we have a spray combustor typically the fuels the liquid sprays accelerated at high velocities to setup this convective enhancement or you can have a case where the oxidizer is being fast over the fuel at very high velocities in order to promote convective burning. But all 3 cases will think about that are very similar in terms of the mechanisms that are involved and in all 3 cases the finer the particle or the finer the droplet, the greater is its propensity to participate in a chemical transformation.

So, in terms of particle characteristics that are very crucial obviously, when you think about a particle that participates in any chemical process, the number one characteristic is its size. The size of the particle determines the rate at which it gets subjected to these chemical processes. The number 2 characteristic is its chemical nature; what is the particle made of and shape actually, plays a less critical role in these particular aspects in terms of the Nusselt numbers. For example, governing dissolution or sublimation or even the kinetics of burning of a droplet, the shape of the material the particle or a droplet actually becomes secondary factor compared to its size. So, clearly among the morphological characteristics size is the most important parameter but, as I was saying second most important parameter is the chemical makeup of the particle or the droplet. That is why it is important to do compositional characterization of a particle or a droplet in order to understand its ability to participate in chemical processes. In the next lecture we will begin discussing methods to characterize the composition of the chemical makeup of particles. Any questions on what we have discussed today? See you in the next lecture then.