

**Mechanical Unit Operations**  
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**Lecture 14 - Size Enlargement Methods**

Welcome to course Mechanical Unit Operations. The title of this particular lecture is Size Enlargement Methods. Till now we have seen the requirement, necessity of the size reduction, how to do the analysis of the size reduction process, what types of size reduction forces are existing, what kind of equipments are available; those things we have seen. We have also seen the storage and conveying of bulk solids, crushed solids and then lumps as well; those things which we have seen.

But sometimes it is also necessary to do a kind of size enlargement for a few type of applications, so that is the reason this particular model we are going to discuss size enlargement methods and then equipment available for the size enlargement of fine particles. So before going into the details of the size enlargement methods, first let us discuss about why this size enlargement is required, under what conditions it is having a kind of important role, is it necessary to do always kind of size reduction, what is the like you know in general size of the particles that they should be enlarged, those kind of things we will see. And then we see the effect of such particles if we do not do the enlargement.

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So why size enlargement required? Because fine particles we understand that there are very difficult to handle and in bulk they do not flow readily, that is what in general we have seen. Even in the previous modules, in the storage and conveying section also we have seen, if the

particles are very fine, what happens? They may be, let us say it is a kind of bin or a kind of hopper is there. So if the particles are very fine, they may be sticking to the wall, right clinging to the wall in general and they may not be falling down comfortably, right?

When this valve is open at the bottom or sometimes they may be forming a kind of bridge, just above the this discharge opening they form a kind of bridge such kind of you know friction they have amongst themselves, so they have a kind of bridging so that whatever the material is there below that bridge formation that may be discharged but above that one that will not be able to discharge and then this bridging is having such kind of strength. So that will not allow the material to flow down. This kind of issues we have seen and then this kind of issues are very much severe especially for fine particles.

Why it happens? Because they have the tendency to adhere together as conglomerates due to a result of action of their surface forces. They have surface forces, surface forces come into the picture especially when the particles goes towards the smaller and smaller size. Very fine particles they have a kind of surface forces like you know they may have the electrostatic charges, they may also have a kind of Van der Waals forces etcetera, those kind of forces they try to make these particles to come together and then form kind of a conglomerates.

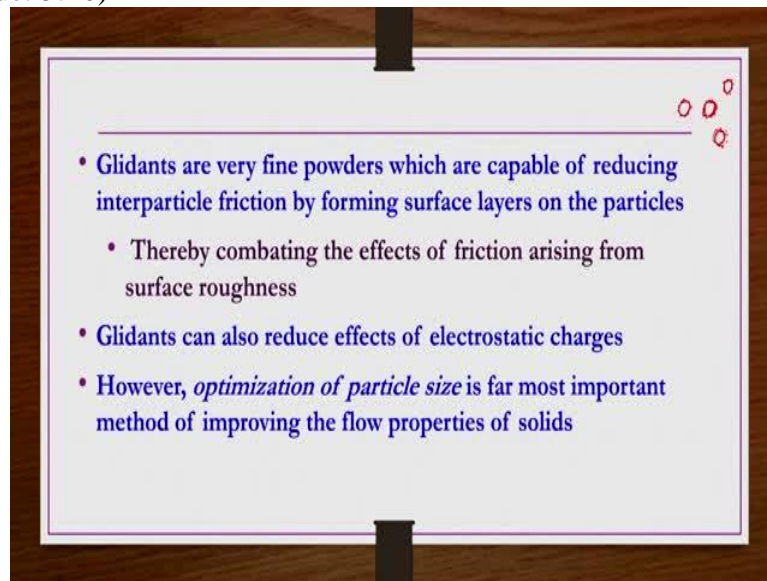
Those surface forces are playing a kind of active role, and then because of the surface forces these fine particles they tend to adhere together to form conglomerates. And then further finer the particles the greater is the specific surface of the particles. So if the specific surface of the particle is large or the particle is very fine that means very small size diameter particle, then in general it will take a long time to settle down. Because of that one you know what happens, these gravitational forces acting on that particle would be very small.

Though the gravitational forces whatever they make this particle to settle, so they may not be settled because of, let us say this room they may be having several fine particles, dust particles, very very small particles which we may not be able to see with naked eyes. But they are suspended, still the gravity is acting but the gravity is so small for those particles that they will not be able to settle down or they settle down in infinite time. Okay, very large time.

So under such conditions what happens? They stay apart from each other during the flow so then they will not be able to transport it easily. Then what is the remedy? A simplest remedy that one can use a kind of a glidant, glidant is a kind of external material that is a kind of fine powder that forms a kind of a layer amongst the around these fine particles and then that will reduce the surface forces like even those some of the glidants they also avoid or reduce the

electrostatic forces or the strength of electrostatic forces so that they can flow freely if the separation or the flow strength is sufficient enough for the flow to occur.

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So what are these glidants? Glidants are very fine powders which are capable of reducing inter-particle friction by forming surface layer on the particles. Okay, so let us say we have a kind of a particle, fine particle like this, like this, so then these glidants they form a kind of layer around the surface of this particle so that you know what happens that whatever the inter-particle friction is there between these particles that will be reduced because of this interfering layer formed because of these glidants.

So that is the purpose of using these glidants, so that this inter-particle friction may be reduced by forming a layer along the surface of the particles. Right, so that the friction arising from surface roughness may be reduced and then accordingly the conglomerates that the particle adhering together that particular phenomena may be reduced. Glidants can also reduce effects of electrostatic charges as well. So not only the surface forces, other surface forces you know reducing inter-particle friction etcetera, they also reduce electrostatic charges as well for such fine particles.

But the problem is about this particle they are much smaller than these finer particles themselves and they are forming a kind of powder layer they are kind of powdered material and then forming a kind of layer. And these materials should have a kind of similar if not exactly same as the ingredient material or active ingredient material let us say, whatever the fine particles that we have we are worried about the transport and handling let us say, those particles let us call the active ingredients. Along those when you take these glidants, mix with

the active ingredients, sorry that is finer particles, so then these glidants may be forming a kind of layer but these glidants should have a similar thermo-physical properties as a kind of active ingredients, right.

And then they should be inert, they should not be reacting or they should not be causing some kind of other kind of physical changes if it all some temperature or is also included in the process, those kind of thing. And then other important thing is that sometimes you need only active ingredients to be participating in the subsequent processes after the conveying let us say.

Let us say there is a reaction, so before the reaction these fine particles are very difficult to flow, so then you mix with the glidant and then after using the glidant you are comfortably able to move these particles to the unit process where the reaction is supposed to take place. But the reaction has to be taken place with a fluid let us say surrounding fluid, heterogeneous reaction is taking place, that reaction has to be taken place with an active ingredient only not with the glidant.

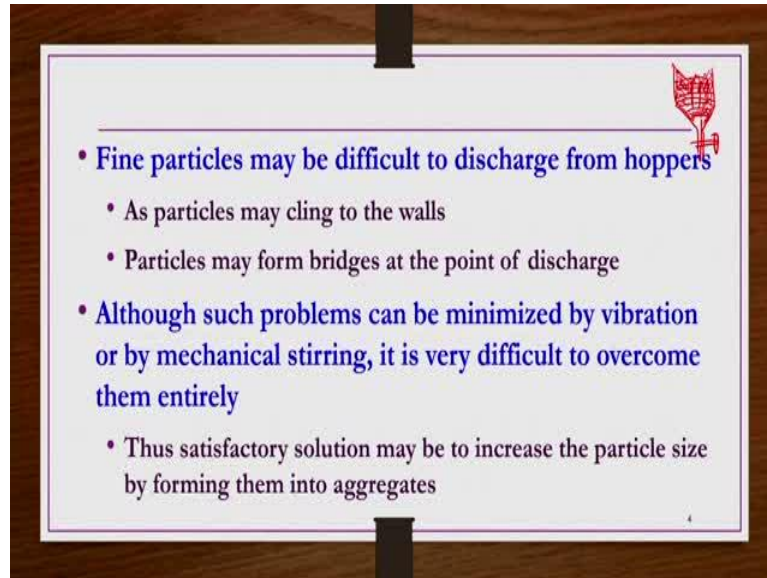
So in such case before the reaction you need to separate out those glidants, that is the another additional problem arising with this glidant. One more issue is that you know you may not find the kind of suitable glidant for all kind of material. For some kind of material, you may find a kind of suitable glidant but finding such kind of glidant for all kind of all other kind of materials is a kind of very difficult task. So that is the reason rather trying to find out the glidant it is better to optimise the size of the particles usable level like you know workable size particles you try to make or have particle sizes of such kind of large size which can be workable which can be easily transportable and which can be easily reactable or undergo some kind of momentum or heat transfer whatever the if there is a reaction whatever the process are there. So that should comfortably take place, that is what is the purpose.

So it is better to have the particles of such kind of size. Because of this reason if you can enlarge the fine particles then they may come to the such kind of certain bigger size and then those size particles may be comfortably undergo subsequent operations.

Right, and then this size enlargement should be very controlled, you cannot have a kind of from very fine particles of microns or some nano size to the almost like you know when you do the enlargement, you may be getting to 1 centimetre 2 centimetre particles such kind of big lumps you are forming, that should not be the case again. It should be controlled process, okay. So that is the reason optimisation of particles size is far most important method of improving the

flow of properties of solids rather than using the glidants because of the reasons that I mentioned.

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Now we have also seen that fine particles may be difficult to discharge from hoppers because sometimes they may be clinging to the wall surface of the container and then forming a kind of bridges as I already drawn a picture and shown a hopper something like this, you may be having fine particles filled over here entirely. So then what happens you know the fine particles may be sticking to the walls, you may not be able to discharge them properly like this.

Right, so like this particles may be sticking to the walls sometimes and then sometimes what happens a kind of rat hole formation kind of thing will be there so that is the material above that discharge rat hole that may be discharging. But the other material may not be discharging because there is a kind of inter-particle friction and that keeps those particles together like this, so they are not flowing. So only that material whatever in the rat hole that is only going comfortably. Sometimes we have also seen that you know the kind of a bridge formation takes place like this, like this bridge formation of the particles will takes place.

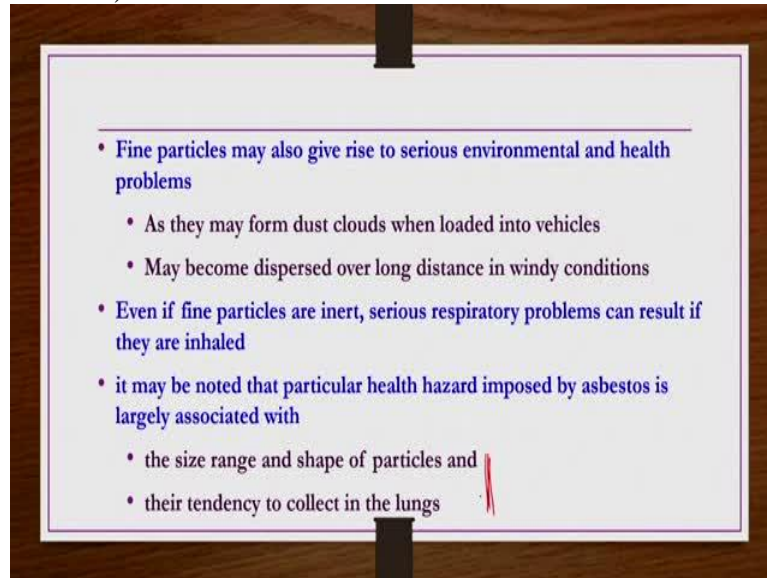
So what happens, the material whatever the material solid fine particles below the bridge formation level is there that will be taken down, that may come comfortably but the material which is above the bridge you know that may not be able to flow down because the particles inter-particle is friction is so high that the particles are coming, adhering together and then forming a kind of bridges and then they are not allowing the material above them whatever is there to flow down.

So this kind of issues are there, so that we have already seen. So as particles may cling to the walls, particles may form bridges at the point of discharge. So although these kind of problems we have already seen that if you provide mechanical vibrations or you know shaking mechanical stirring or you know vibrations etc along the walls of this container or the silo or the hopper, so then it is possible that you may reduce such kind of effects of bridge formation or you can reduce the influence of the rat hole formation, you may even reduce the particle stinging particles you know sticking to the wall, clinging to the wall, those kind of things also you can reduce but you cannot completely avoid such kind of phenomena by using mechanical vibrations or mechanical stirring kind of thing.

So because of that one it is always possible to have a kind of a solution may be to increase the particle size by forming them into aggregates, that is a kind of the best solution best possible, if not the best satisfactory solution, that is going to be a kind of satisfactory solution where you can conveniently move or the where you conveniently make solid particles to move conveniently or flow conveniently, that you can make sure with the kind of satisfactory kind of thing if not by if it is not the best option, it is a kind of satisfactory option.

So increasing the particle size by size enlargement method is a kind of going to be very useful. However the problems associated with the fine particles we have seen till now, are only associated with their flowability kind of issues only but in general they may also cause some kind of health issues as well. That we see now.

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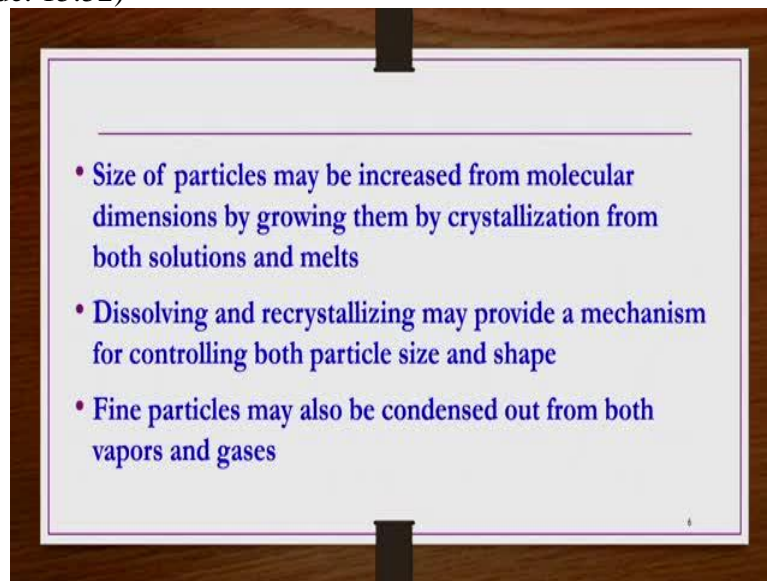
Fine particles may also give rise to serious environmental and health problems. What happens let us say when you are loading or unloading vehicles which is having like you know these fine particles let us say fine particles you are loading in a vehicle or already fine particles are loaded in the container and then you are taking out, unloading it. Then during that process what happens is fine particles may form a kind of dust cloud in the surrounding. So that is very dangerous and then surrounding weather if it is in a kind of windy condition, weather is windy weather then this dust cloud may be carried to the long distance and then that may be causing health problems to the not only the people working in the industry, in the process but also nearby surrounding society as well that is going to affect.

Right, so that is the problem. So in such cases what happens, even if these fine particles are inert so when you inhale them even if it is inert that is going to sting onto the lungs and then that may be causing some kind of respiratory problems, that is a kind of major issue. Now when they are non-reacting when they are inert and then when you are inhaling them by you know because the weather is surrounding air is contaminated like that, then they are sticking to the lungs and then you know creating respiratory problem.

Now you can imagine if these particles are reactive also, if they may also cause some kind of physical or chemical changes inside the body then you cannot even imagine what kind of health issues you know arise. So such is the kind of dangerous problem about these fine particles especially concerned with the environment and health problems. See as they may form dust clouds when loaded into the vehicles may become dispersed over long distance in windy conditions.

Even if fine particles are inert, serious respiratory problems can result if they are inhaled. It may be noted that particular health hazard imposed by asbestos is largely associated with the size and shape of the particles and their tendency to collect in the lungs. Because of these reasons these asbestos particles in general which are you know inhaled because of the such kind of environment in the working place, you know they are causing respiratory problem because of their size and shape and then their tendency to collect in the lungs. So this is other kind of problem of handling very fine particles.

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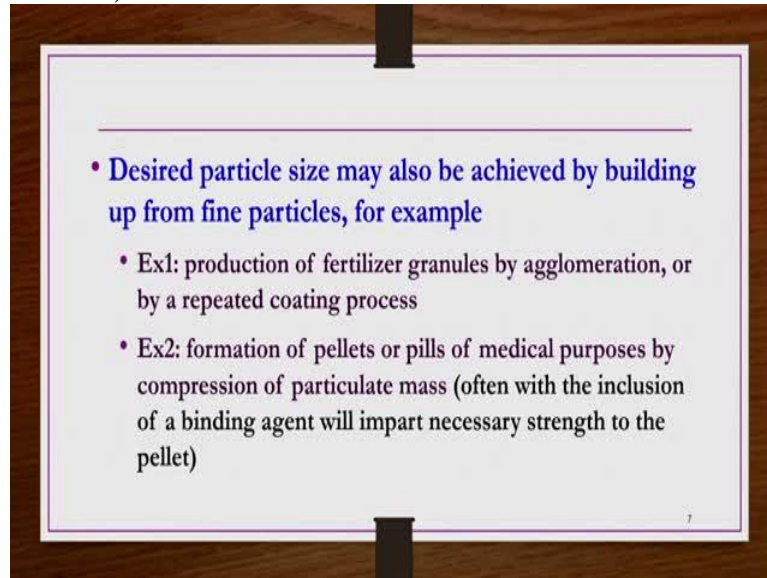


So size of the particles may be increased from molecular dimensions by growing them by crystallisation from both solutions and melts. What you can do, you can find out a kind of solvent in which these particles may be dissolved, so then you dissolve them and then you re-crystallise them so that you know these finer particles initially you dissolve but when you do re-crystallisation you may not be getting such fine particles, you may be getting such a you may be getting slightly bigger particles. So dissolving and re-crystallising may provide a mechanism for controlling both particle size and shape as well.

So fine particles may also be condensed out from both vapours and gases before letting out the industrial effluence let us say industrial effluence gases or vapours whatever having you know particles or particle laden vapours or gases rather just releasing them from the industries before releasing them one can condense them and then collect a kind of bigger particles and then only cleaner gases or vapours should be released out.



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Desired particle size may also be achieved by building up from fine particles, such as like you know production of fertiliser granules by agglomeration or by a repeated coating process. Fertilisers in general we do a kind of agglomeration, the fertilisers also they need a kind of specified size, you cannot have a very fine particles very small size of the fertilisers because they are in general used in the kind of farming etc. And the field you know when somebody spraying this you know fertilisers if they are very small and then surround catching up in the surrounding areas, so that is going to be a dangerous to the people working around, so that way also the fertilisers needs to be of specialised sizes.

They cannot be very fine, very small, very fine size. Then formation of pellets or pills of medical purposes by compression of particulate mass, so sometimes you know especially in pharmaceutical industries we see tableting etc. Tablets consist of so many several particles. So one cannot take those particles as it is, so then a kind of briquetting tableting is done by the compressive forces, compressive action, that is one of the you know size enlargement method that we are going to see anyway. So here we can have a kind of binding agent you know along with this particles so that binding agent will give strength to the material and then the tablet one can have.

Right, so this will also add a kind of addition like you know tableting you know certain exact how many grams of the material should be present in the given or how many grams or milligrams of the active ingredient should be present in the given tablet, those kind of thing controlling and metering can also be done by exact pelleting or briquetting of these tablets.

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So then what is size enlargement? So we have seen already several reasons why size enlargement should be done, there may be many other reasons also, so we have now enough number of reason to study why size enlargement is required, so first let us see what is size enlargement. It is any process whereby small particles are agglomerated or otherwise brought together into larger relatively permanent masses in which original particles can still be distinguished. Okay so that is what like you know when you do this you take few particles and then you do a kind of agglomeration process so that you can have a kind of bigger particles bigger size particles you can have comprising of such small particles.

But they should not be very loose that you know when you give a kind of small forces these particles may be you know this you know and large particles may be disintegrating immediately. But they should not be very strong that if you wanted to disintegrate them then then you need to give a kind of additional forces extra forces kind of thing. They should be relatively permanent masses. Let us say you have taken tablet, so the particles are kind of you know active ingredients but you are adding some kind of binding agents so that you can easily make a kind of tablet which is having sufficient mechanical strength.

But the mechanical strength is so high that even when you inhale it takes several hours to dissolve inside your stomach to so that the active ingredients should come out and then be active and then show its affect on the kind of a disease, so it is not going to work, it should be dissolved it should be disintegrated as early as possible. So those kind of things should be taken care. So that is the reason size enlargement is any process whereby small particles are

agglomerated or otherwise brought together into larger relatively permanent masses in which original particles can still be distinguished.

So they should not be very loose also. If they are very loose by the from the point of production to the consumer point it reaches all the particles then enlarged particles may be disintegrated and then smaller particles may be forming, so that is not going to lose either way, it should be controlled properly. So where do we find such kind of size enlargement in general? We find in pharmaceutical and food processing industries, we find in consumer products specified size and shape of the products are required so that is what we call consumer products.

And in fertilisers and detergent productions, detergents also need to be in the kind of specified sizes, they cannot be very fine. Otherwise they may cause a kind of environmental and health hazardous to the people using them. Then mineral processing industries et cetera where we use in general the size enlargement methods. So agglomeration is the formation of aggregates through the sticking together of feed and or recycled material. So whatever the material that feed material is there, you sticking together and then form a kind of certain bigger particles. That is what agglomeration.

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So now what should be the objectives of size enlargement in general? So let us say in agricultural chemical granules or pharmaceutical tablets the objective should be they should provide provision of a defined quantity to facilitate dispensing and metering. So in a given size of the tablet the amount of the active ingredients and then the amount of the binding material, binding agent will be very much defined and then properly control, so that provision you will be able to have if you are doing this you know size enlargement and tableting kind of thing or

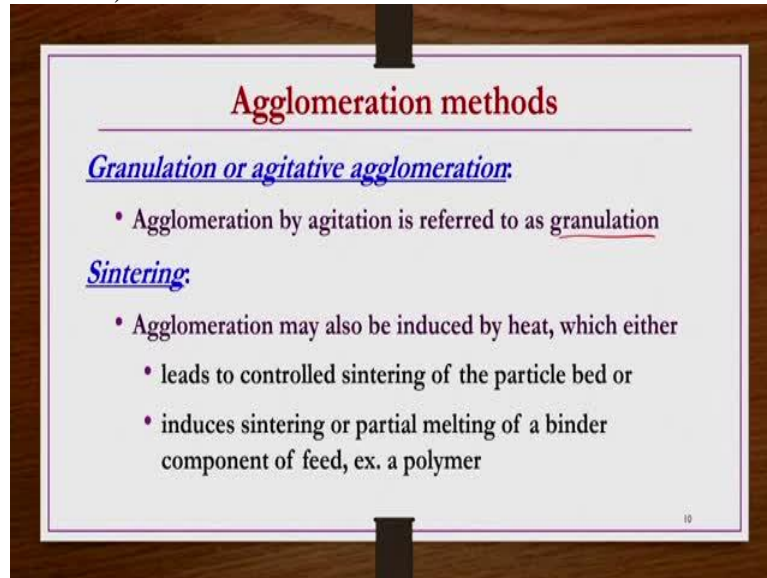
you should do the size tableting kind of process such a way that in a given tablet you should have that much milligrams of active ingredients only.

You cannot afford to have a variation or change in active ingredients composition from one tablet to other tablet. So that kind of provision of a defined quantity to facilitate dispensing and metering of this tablet etc. Then improved flowability also, so when they are after forming by some briquetting operation or compressive kind of size enlargement methods whatever the tablet that you bring, you will not be packing immediately in the wrappers.

So you may be flowing from one process to the or the product collection vessel, so the flowability should be comfortable and then during the flow process they should not break etc, those kind of things. So that is improved flowability should be there and then creation of non-segregating blends of powder ingredients. Briquetting of waste fines such as elimination of dust handling hazards or losses etc, then granulation of fertilisers to reduce caking and lump formation. In general in fertilisers what happens if the particles are very small, so actually you know the process you will be doing some kind of you know drying process also.

So they if the process is not done properly, a kind of caking or lump formation kind of thing will also be take place in general. So when you do the granulation of these fertilisers one should be careful that they not forming cakes or lump kind of thing or that by size enlargement such kind of existing caking lump formation should be reduced. And then increased bulk density for storage and tableting of the feed's etc. Then control of solubility instant food products and so on. So like that if you application wise if you see their objectives also keep on changing and then slightly modifying from one to the other case. So these are the objectives with reference to some kind of specific applications of the size enlargement.

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**Agglomeration methods**

Granulation or agitative agglomeration:

- Agglomeration by agitation is referred to as granulation

Sintering:

- Agglomeration may also be induced by heat, which either
  - leads to controlled sintering of the particle bed or
  - induces sintering or partial melting of a binder component of feed, ex. a polymer

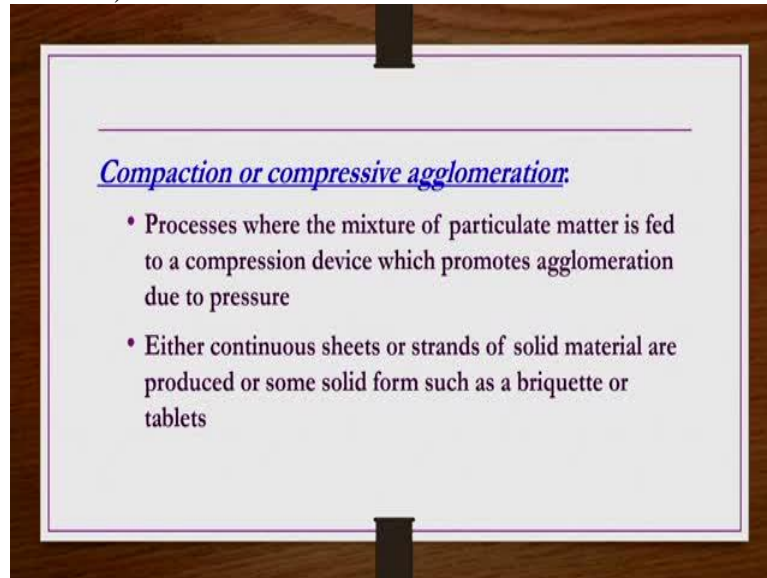
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Then agglomeration methods, so what we understand agglomeration is a kind of sticking together smaller particles, fine particles and then trying to form a kind of bigger particle. So now how does it happen? What kind of methods are available? That is what we are going to see. Primarily there are two methods only but we can have a kind of third method also. So we see these three methods. Like first one is the granulation or agitative agglomeration. Here agglomeration by agitation is referred to as kind of granulation.

Here what we have, you have a kind of cylindrical vessel where you know a drum kind of thing you are having so in which you take the feed includes like you know active ingredients, solid fine particles along with the blend particles et cetera, so those drums rotate once you take this material inside and then agglomeration you know bigger size particles formation takes place. This process can be continuous or you know batch wise as well.

So here what happens, the mechanism primarily is the agitation. So whatever the agglomeration is occurring by agitation is referred to as a kind of a granulation process. Then sintering, agglomeration may also be induced by heat, which either leads to controlled sintering of the particles bed or induce sintering or partial melting of a binder component of feed. Binder components of a feed in general are kind of polymer solutions or some kind of taste inducing agents or something like that. That is what is the sintering process.

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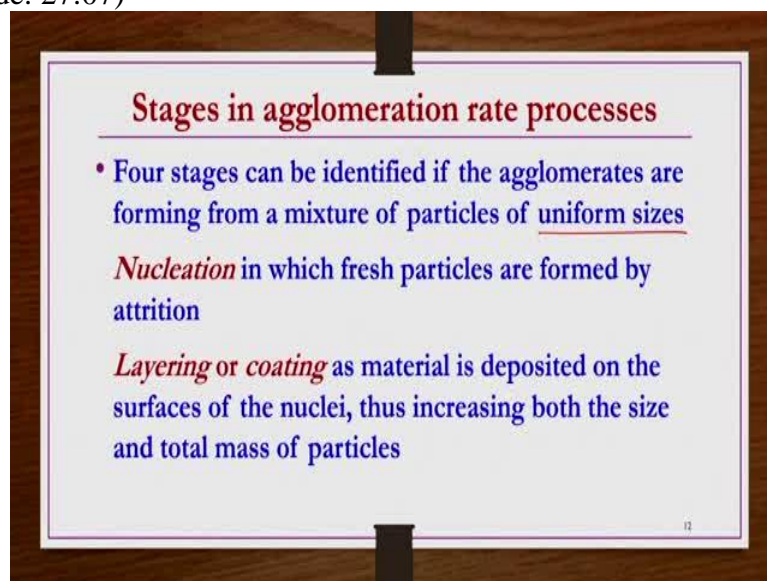
*Compaction or compressive agglomeration:*

- Processes where the mixture of particulate matter is fed to a compression device which promotes agglomeration due to pressure
- Either continuous sheets or strands of solid material are produced or some solid form such as a briquette or tablets

The third one is the compaction or compressive agglomeration. Here what happens, the processes where the mixture of particulate matter is fed to a compressive device which promotes agglomeration due to pressure. This is in general used for the kind of briquetting or tableting kind of thing where you take the fine particles and then you apply compressive forces so that they form a kind of tablets. So either continuous sheets or strands of solid materials are produced or some solid form such as briquette or tablets.

So these are the three agglomeration methods in generally one can take, primarily actually two only; granulation, the other one is the compaction but sometimes sintering is also being used for agglomeration.

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**Stages in agglomeration rate processes**

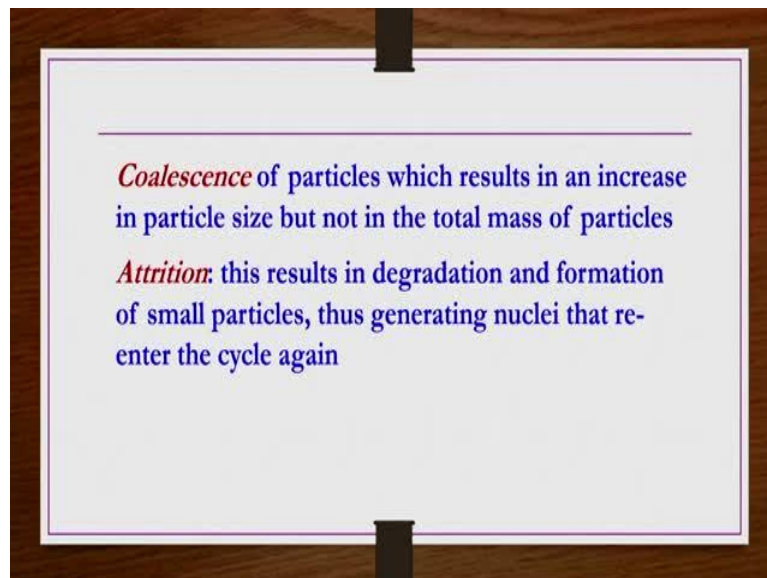
- Four stages can be identified if the agglomerates are forming from a mixture of particles of uniform sizes
  - Nucleation* in which fresh particles are formed by attrition
  - Layering or coating* as material is deposited on the surfaces of the nuclei, thus increasing both the size and total mass of particles

12

Then stages in agglomeration rate processes, what stages in general you have? In general there are four stages are possible. Four stages can be identified if the agglomerates are forming from a mixture of particles of uniform sizes. That is uniform mixture that you have, so from that uniform mixture of fine particles if you take and then you do a kind of agglomeration so you may find the agglomerates in four stages. What are those stages? We see now.

First one is the nucleation in which fresh particles are formed by attrition. The next is the layering or coating as material is deposited on the surfaces of the nuclei, increasing both the size and total mass of the particles takes place. Okay, a kind of layering or coating of the fine particles will be done by this layering or coating mechanism or this step.

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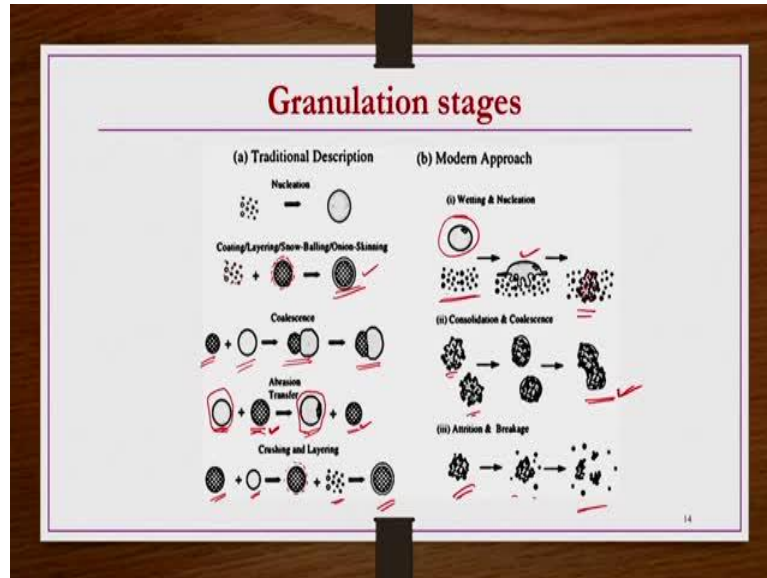


Then is the coalescence, coalescence of particles which result in an increase in particle size but not in the total mass of particles. Whatever the smaller size particles are there they come together and then coalesce and then they form a kind of bigger size particle. Right, but the size changes the size increases but the total mass of the particle does not change. Whatever the let us say you take two particles only, whatever the weights of the individual particles are there if you add together that should be the weight of the agglomerated bigger particle forming by the coalescence.

Okay, sometimes the entire two particles complete material may not be forming a kind of bigger material, so one bigger particle and then some fracture fractures may also forming, so that entire mass will remain same though the bigger size particles are forming. Then attrition, this results in the gradation and formation of small particles, thus generating nuclei that re-enter the

cycle again. So those are the different stages that one may come across in kind of agglomeration.

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So granulation stages, we have seen till now stages in agglomeration, now we see stages in granulation. So here we see this kind of pictorially, here in a traditional description what we have, we have a small nuclei, small kind of particles. So they join together because of the nucleation and then they form a kind of particle like this. Let us say that particle is like this and then you have a kind of a binding agent or something like that the small particles here. So these particles will form a kind of a layer along the surface like this.

Or a kind of you know coating will take place, so this is known as the coating or layering or snowballing or onion skinning kind of names are given. So then you have a particle bigger particle like this. Then coalescence is also one of the step stages, so here let us say this one small particle, this one slightly bigger particle they join together and then they form a kind of bigger particle like this. They even come closer and then form a bigger particle like this as well. Then there is also a kind of abrasion transfer, so let us say here now you take two same sized particles, so then in this abrasion transfer what happens, some of the material goes in and transferred to the other particle.

So then one particle, one of this particle becomes slightly bigger, so this particle is now slightly bigger compared to this particle because somehow this material is transferred into this material by the abrasion. So the obviously the remaining material remaining other particles, that size should be decreased. So now you can see this shaded particle this particle the size is decreased



compared to its previous size before the abrasion transfer. So this is what is the abrasion transfer.

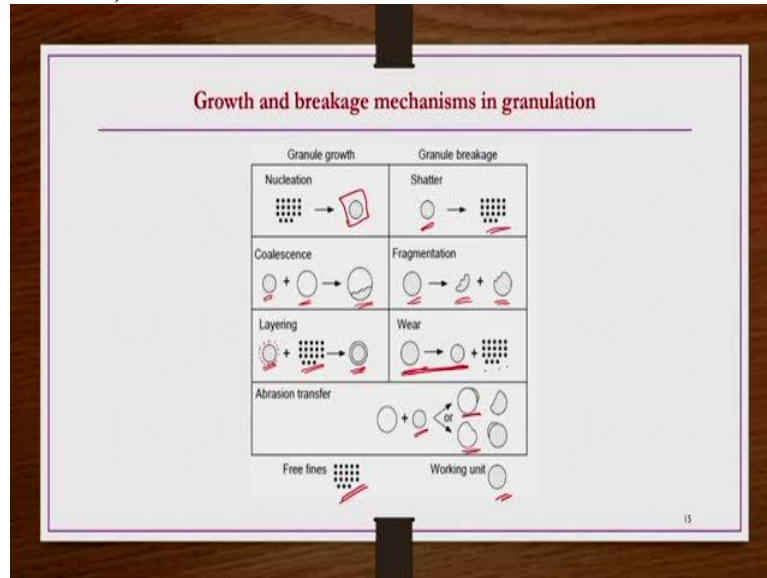
Then crushing and layers happening together. Here you know particle is there and this particle may be undergoing a kind of crushing by the attrition or something kind of those kind of processes and then this will form a kind of a after you know crushing these smaller particles forming a kind of layer along this you know the other particles so that the altogether bigger particle you know formation takes place because of this enlargement. Right, so these are the steps in general considered in traditional granulation process.

But nowadays in modern processes it is done slightly different way also, whatever the small particles nuclei kind of things fractures are there, so here wetting and nucleation takes place, so then some kind of solvent you can take or slurry you can take or you know some kind of let us say binding agent you can say so that when you mix here they are forming like this. And then further these particles may join together with the help of the additional fluid medium that you have taken and they may be forming a kind of slightly bigger particle like this though there may be some smaller particles still remaining.

Right, so such kind of bigger particles then they can consolidate and then coalesce, they can undergo a kind of coalescence and then they can form a kind of a bigger particle shown like this. Or this particle whatever this you know after wetting and nucleation or even after consolidation these particles may also undergo some kind of attrition because this process you know definitely the interaction between the particles will be there, so then there will be a kind of attrition amongst those particles. So then because of that one some fraction of this material may be disintegrated and then some small particles are forming.

If the attrition is more so then you know many smaller particles may be forming like this. So that is what attrition and breakage. So these are the granulation stages in general both traditional approach and modern approach.

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Next is the granule growth and breakage mechanisms in granulation that also we see. This is a kind of mechanism which has been you know taken from a reference book where it was shown as a kind of steps for their optimisation problem. This is not truly from a kind of experimental work but optimisation problem this is what we have taken that, okay. So growth and breakage mechanisms in granulation. Here granule growth how it takes place? If you have this smaller nuclei something like this and then you allow this nucleation process to take place, so let us say the particle you may be forming like this. Now such kind of bigger granules that you have, so you may be having different size of granules.

Let us say you have smaller granule and then bigger granule, so then they may coalesce and then they may form a kind of further bigger granule and they not necessarily kind of spherical, they have been taken in a spherical shape just for the kind of reference so that we can see, understand easily here, okay. So there is another process layering, so let us say one granule is there, small size granule that you found but there are nuclei available, so they may be forming as a kind of layer along this surface of this smaller granule and then you can have a kind of layered granules which is having size slightly bigger than the its initial size.

So this can also be layering is also a kind of a granule growth stage mechanism, one of the mechanism. Now granule breakage mechanism how does it take place? Let us say you have already granulated bigger size particle, now you shatter it, then you form a kind of nuclei like this. It is just a kind of reverse of the nucleation process. Then fragmentation let us say you have a bigger size granule, when you do the fragmentation you can have a kind of two different

sized you know granules, different shape and different sized granules you may get because of the fragmentation and there may be also be a kind of wearing.

So when you do this wearing, the shape may be remaining same but the size reduction takes place because of this one and then so your smaller granule will take form and then along with this there will be some kind of nuclei will also form. In the abrasion transfer we have already seen, so we have one bigger granule and then one smaller granule is there, so either of these two processes may take place. The smaller granule some of the material of the smaller granule may transfer to the bigger granule or attached to the bigger granule and then kind of bigger further bigger granule may be forming because of the abrasion or otherwise the somehow the material from the bigger granule may be coming and attaching to the smaller granule because of the abrasion and then there may be a kind of intermediate sized particle may be forming like this.

So here that is what, other things like these fines, the dotted ones whatever shown in this picture are nuclei kind of, there are very very fine small particles, they are very difficult to handle. So they are not of workable size, they are not in workable size. So then workable sizes in general like you know bigger like you know circles et cetera shown here, so they are working units kind of things, such kind of enlarged particles are better to handle and then transport. This is about the growth and breakage mechanisms in granulation. Now we see some details about the agitative granulation or agglomeration and then compressive agglomeration and those some details we see now here.

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### Agitative granulation

- Feed is introduced into a vessel along with granulating medium and agitated to get granulated product
- This process include fluid-bed, pan (or  $X=(x_1, x_2, \dots, x_n)$ ), drum and mixer granulators
- This also used as coating operation for controlled release, taste making, etc.
- Feed typically consist of a mixture of solid ingredients (fine particles), binders, diluents, disintegrants, flow aids, surfactants, wetting agents, lubricants, fillers, or end-use aids such as colours, taste modifiers

The diagram illustrates the agitative granulation process. It shows a 'Process vessel' where 'Feed' enters from the left and 'Product' exits to the right. Above the vessel, the input is labeled  $F=(e_1, e_2, \dots, e_j)$ . Below the vessel, the output is labeled  $Y=(y_1, y_2, \dots, y_n)$ . To the left of the vessel, a graph shows a distribution curve for 'x' with a peak at  $x$ . To the right of the vessel, a graph shows a distribution curve for 'y' with a peak at  $y$ . Below the vessel, text indicates '(x or y) = (size, coating, strength, surface properties, quality metric)'. The number '16' is visible in the bottom right corner of the slide.

So agitative granulation in general we have a kind of a vessel in general a kind of a drum. It may be having disk, and then mixer and all those things. So here let us say you have a feed, this feed is a kind of small size particles, very small size particles but uniform mixtures something like this, very fine particles. These fine particles along with some kind of binding agents are kind of size enlargement medium, they will be taken in a kind of process vessel.

Or after taking into the process vessel you can spray some kind of solvents or slurries onto this bed of particles and then rotate this drum so that agitation may take place and then because of that one you know because of that agitation these particles you know stick together because of this sprayed slurry or solvent we have whatever whichever we have taken. Because of that one they will attach together themselves and then they will form a kind of bigger particle something like this.

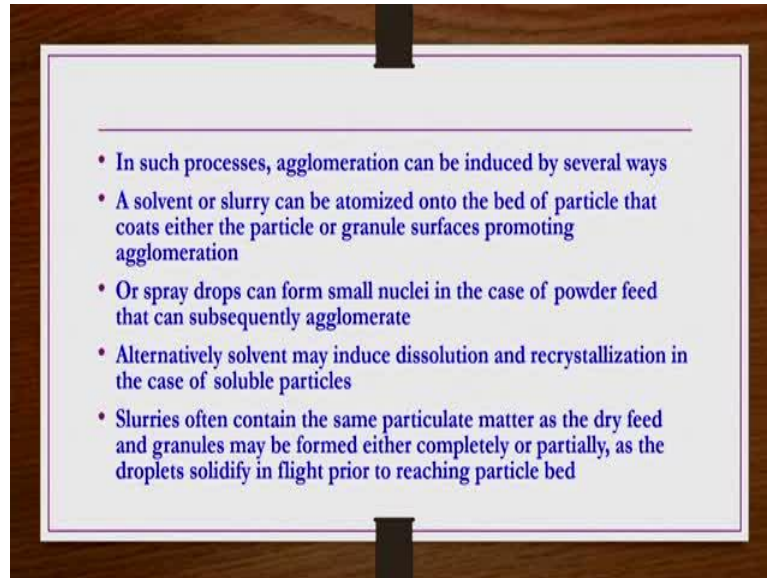
So which are in a kind of better workable size rather than here, such feed if you see the size of the each particle is very small, it is not one, feed is not one single particle shown here, it is a kind of having so many fine particles as dotted particles like this here. When you do the agglomeration they are all forming kind of you know lumps kind of thing, agglomerates kind of thing here. Because of the agitation they have formed and then this particle is a kind of in a better size to handle in subsequent in a given transport operation. So here process variables for the feed let us say X and then for Y it is product let us say Y, right.

This vessel E is a kind of some kind of process stages that are going inside this equipment, so it is a kind of generalised picture only. Okay, here what happens in general? Feed is introduced into a vessel along with granulating medium and agitated to get granulated product and this process include fluid bed, pan or disk, drum and then mixer granulators. This also used as a coating operation for controlled release, taste making etc. Then feed typically consist of a mixture of solid ingredients that is fine particles or the active ingredients that I have been telling those fine particles.

Then some kind of binders, they are something like a polymer solution or some kind of solvents or some kind of a slurries et cetera. Diluents may also be there, disintegrants may be there, flow aids may be there, surfactants may be there, wetting agents may be there. Lubricants, fillers or end-use aids such as colours, taste modifiers et cetera, may also be there. It is not necessary that all of them may be there in each and every process, it is not true. Depending on the applications you know some of them may be there, some of them may not be there. Depending

on case to case you may need you know some of them or sometimes you may need all of them but it is not true that always you need all of them.

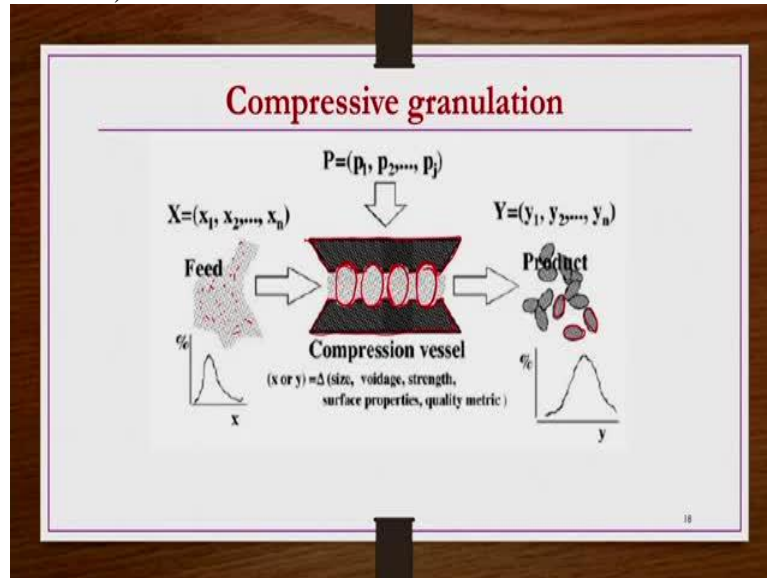
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So in such processes agglomeration can be induced by several ways, one way is a solvent or slurry can be atomized onto the bed of particles that coats either the particles or granule surfaces promoting agglomeration. Or spray drops can form small nuclei in the case of powder feed that can subsequently agglomerate. Or alternatively solvent may induce dissolution and recrystallization in the case of soluble particles. If the particles are soluble in a solvent which we have taken then the particle may be dissolved so that a kind of solution or slurry it may be forming.

That solution or slurry if you recrystallize then you may be having a kind of bigger size particle rather than having a kind of fine particles as initially that you had. So dissolution and recrystallization may also be one kind of a way that is causing this size enlargement. Any of these methods may be possible to have this size enlargement. Slurries often contain the same particulate matter as the dry feed and granules may be formed either completely or partially as the droplets solidify in flight prior to reaching particle bed. Now this is about the agitative granulation, now we see compressive granulation how to do that one?

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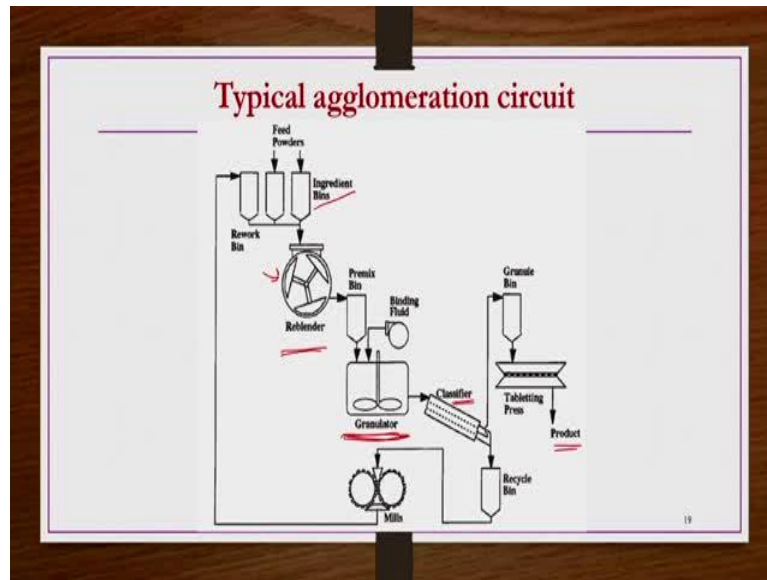
Compressive granulation in general what we have, we have a kind of a setup like this, so this let us say two plates are there here, not kind of plates, sections are there like this. So the fine particle whatever the feed fine particle, very small size particles are there they themselves as individual without any addition may come here, may be taken inside this compressive device. And then you apply compressive forces so that they may come together and then form a product like a tablet of this shape, whatever the shapes that we have.

You know this kind of shapes for tablets we may get in general. So sometimes it is not possible if you take only feed material and do compressive action. If you apply it may not be possible that you may get the required product size. So then under such conditions the feed may be added with some kind of binder material or the you know taste modifier et cetera, as per the requirements and then they pass through this thing. So when they are passing through this one, these sections these two sections may come close together because of the compressive forces or when the compressive forces are applied.

And then the enlarged particle which are now product they may be coming as a product. Now here you can see, here also initially very fine particles are there, after that you have a kind of particles slightly bigger sized which are in a kind of workable size, they can be easily transported, they can be easily you know undergo reactive process whatever it is. They can easily be disintegrated as well by dissolving or by applying some small strength without applying high-strength, you can apply very less strength and then you can get you know disintegrating these products either way.

So that is they are large masses but relatively permanent masses, they are not you know permanent masses, that means you can disintegrate them as well. So this is about the compressive granulation.

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So now typical agglomeration circuit. If you take any agglomeration section or circuit there may be several operations involved, several operations involved and then sooner when we see the schematic we can understand that whatever the agitative granulation is there or agitative agglomeration is there, that is a kind of intermediate rather than a kind of final product. Whereas the product that you get from the compressive units that is a kind of a final product. So that is what we see now here.

Let us say typical agglomeration circuit looks like this, not necessarily that this is how each and every agglomeration unit will be there, there may be additions, there may be, there may not be this many units involved, these many unit operations may not be involved in a given agglomeration circuit or in some other agglomeration unit much more number of unit operations may be involved and shown here. So anything is possible but this is just a kind of a typical agglomeration circuit. So here what we have, we take this feed powders along with the ingredients from the ingredients bin in a blender in a blender like this here. So this is rotates this blender is having a kind of you know agitative provision.

So when it rotates agitations will take place. So from here you take that material to premix bin and then again at the binding fluid here and then you do a kind of mixing so that granulation takes place. So that the kind of granulation takes place, so after here whatever the material that granulated material is there, that you pass through classifier or you pass through a kind of

screen so that you can separate the undersized oversized material whatever the product that oversized material, now here the oversized material should be a kind of product in general because we are doing a kind of size enlargement, they may be taken to the other granule bin.

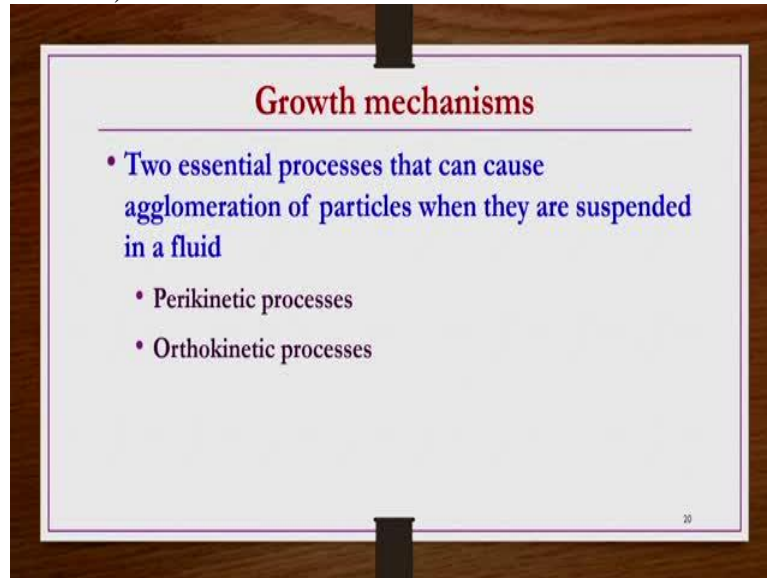
From there we may be taking them to a kind of compressive device where tableting may be done by the pressing operation, compressive operation and then final products may be coming as a kind of tablet or briquetting. Whereas the undersized material or the material which are small enough and which may not good enough to undergo kind of briquetting or tableting kind of operation, they may be taken to the recycle bin. From the recycle bin they may be taken again to the mills, they may be disintegrated using this smaller strength mills and then they may be taken back to the kind of a recycle unit where the circuit continues.

So this is the kind of typical agglomeration circuit where here now what we are seeing that you know this is a kind of agitative granulation step here from here to here. So here whatever the product is coming that we are now taking as a kind of a final product, after this we are doing a kind of classification or using the classifier, classifier in the sense we are doing the size separation. So size suppression we do and then the particles which are of the suitable size they may be taken to the kind of compressive device where the compressive agglomeration takes place in order to get the product in a kind of tablet form.

Whatever the material after the classifier which is not found good enough for the compressive action they are further taken to the recycle bin, from the recycle bin they can be taken in a kind of small strength mills where these particles because after granulation they may not be suitable here, that is the reason they are brought to the recycle bin but they are enlarged. They are enlarged, so those enlarged particles will again disintegrated using these mills and they will be fed back to the feed powders tanks and then that process continues until the entire material has been converted as per the required size.



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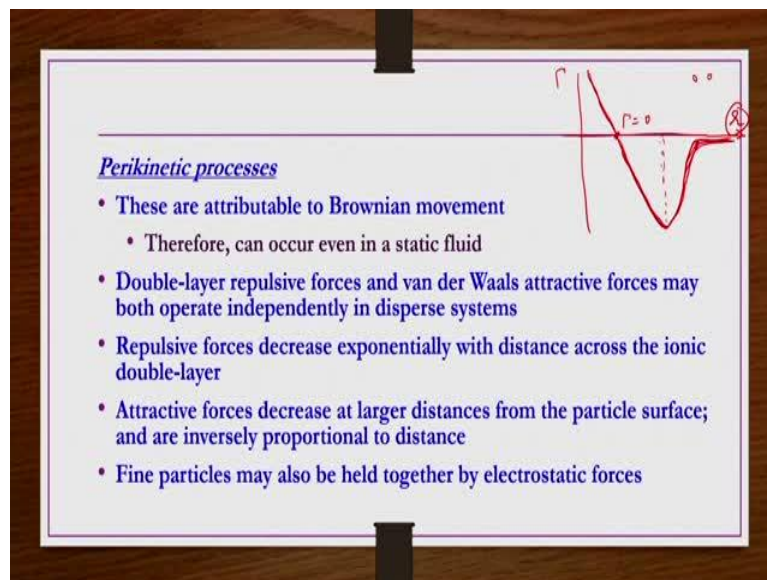
**Growth mechanisms**

- Two essential processes that can cause agglomeration of particles when they are suspended in a fluid
  - Perikinetic processes
  - Orthokinetic processes

20

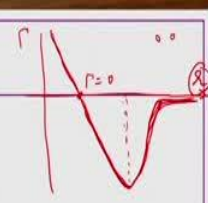
So what are the growth mechanisms? Growth mechanisms, two essential processes that can cause agglomeration of particles when they are suspended in a fluid rather than in a kind of individual solid phase. When you suspend these fine particles in a fluid then two essential processes may take place, they are nothing but perikinetic processes and then orthokinetic processes.

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**Perikinetic processes**

- These are attributable to Brownian movement
  - Therefore, can occur even in a static fluid
- Double-layer repulsive forces and van der Waals attractive forces may both operate independently in disperse systems
- Repulsive forces decrease exponentially with distance across the ionic double-layer
- Attractive forces decrease at larger distances from the particle surface; and are inversely proportional to distance
- Fine particles may also be held together by electrostatic forces



So we see individually, perikinetic processes, these are attributable to the Brownian movement, that is you know even when you have a kind of stationary fluid without any bulk motion, so at the molecular level, the molecular vibrations, interactions, molecular rotations et cetera, those kind of things will be there. Those kind of vibrations, rotations of the molecules induce some kind of motion, that is known as a kind of Brownian movement or the Brownian motion. So

these are whatever this perikinetic processes are there, they are attributable to Brownian movement, so they may be existing even if there is no bulk flow.

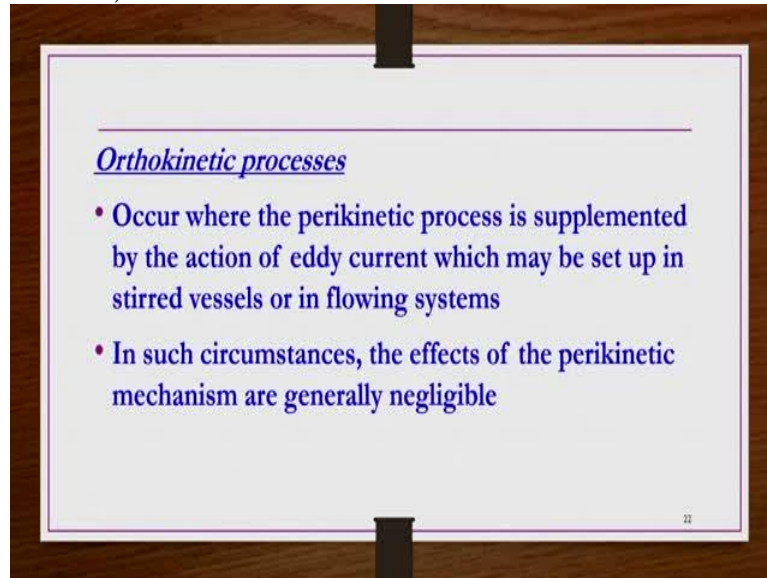
Though these particles suspended in a fluid but the fluid is in a kind of stationary position stage, so then under such conditions also these perikinetic processes will occur because they are attributed attributable to the Brownian movement. So they can occur even in a static fluid. For the double layer repulsive forces and Van der Waals attractive forces may both operate independently in dispersed systems. Repulsive forces decrease exponentially with distance across ionic double layers. Attractive forces decrease at large distance from the particle surfaces and are inversely proportional to distance. Fine particles may also be held together by electrostatic forces.

So now coming to these forces attractive and repulsive forces, let us say we have a kind of Y axis which indicate the interactive forces, interaction forces between these particles and then we have a kind of X-axis that indicates a kind of distance between the particles  $R$ . So these forces you know repulsive forces are acting like this, something like this, so these forces decreases and then decreases may not be a kind of it is a kind of this repressive repulsive forces kind of exponentially decreasing with the distance like this. Then let us draw the figure first.

So these are the repulsive forces, they are decreasing and then at this point what happens, the interaction force becomes 0. There is no interaction between the particles and after that you know when you further increase the distance between these particles, kind of attractive forces increases up to this point certain  $R$  value. And then further if you take these particles away from each other, so what happens? This attraction force started decreasing and then there will not be any kind of attraction forces, attraction forces after certain distance.

This distance is between these two particles and then this small this itself the particles are in a kind of micron size and then this distance  $R$  is also in small dimensions in some micron size. So this is how this interaction forces in general may be there. So under these conditions you know where these kind of forces are existing, so all those cases under those circumstances we have this perikinetic processes are having a role.

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Whereas the orthokinetic processes which occur where the perikinetic process is supplemented by the action of eddy current which may be set up in stirred vessel or in a flowing system. If the fine particles along with a fluid you may be taking in the kind of vessel and then stirring them rigorously or you taking or the flow is a kind of very high flow so that you know eddy currents may be forming, something similar to the kind of turbulence etc.

So under such kind of eddy currents presence the influence of the Brownian movement may be very small or in comparison to these eddy currents which are occurring because of the rigorous stirring or the high velocity flow system, so in comparison to them the Brownian movement may be very small. So when orthokinetic process is dominating, the contribution from the perikinetic process may be very small. Both of them may be there but the contribution may be very small.

Let us say if the fluid is almost like under stationary or if it is moving with very very slow velocity, very small velocity something like  $10^{-6}$  meter per second or  $10^{-10}$  meter per second, such small velocity, then there is a bulk flow though it is a very small. But the flow is bulk flow is very small. Under such conditions Brownian movement or the process which is occurring because of the Brownian movement that is more important or having the lead role compared to the bulk motion.

So that is what happens in the perikinetic process. Orthokinetic process it is different way, bulk motion is so strong that the Brownian movement or the contribution from the Brownian movement can be negligible or considerably very small compared to the orthokinetic processes. In such circumstances where the stirring of the vessel having these contents of solid and fluid

stirring them or system is in rigorous flow, under such conditions the effects of the perikinetic mechanisms are generally negligible and orthokinetic processes dominate.

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• For perikinetic processes:  $d_t^3 = A_1 + B_1 t$  ✓

• For orthokinetic processes:  $\log\left(\frac{d_t}{d_o}\right) = A_2 + B_2 t$  ✓

• These equations are valid for initial stages of agglomeration, otherwise they would indicate an indefinite increase of size  $d_t$  with time  $t$

- $d_t$ : agglomerate size at time  $t$
- Dimensions of  $A_1$ :  $L^3$   $A_2$ : dimensionless,  $B_1$ :  $L^3 T^{-1}$   $B_2$ :  $T^{-1}$

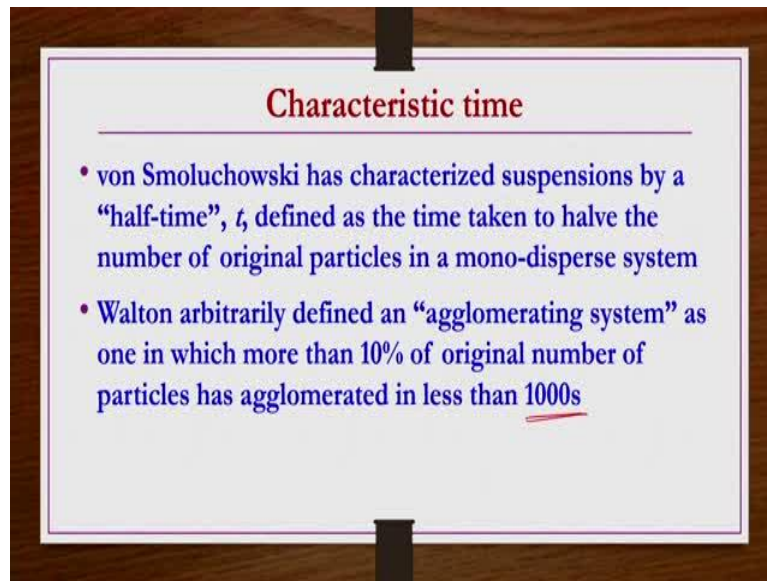
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For perikinetic processes this size of the particle let us say after time  $t$  if you wanted to increase it to the size  $d_t$  size, so then  $d_t^3 = A_1 + B_1 t$ , that is suitable equation to represent this perikinetic process in general. For orthokinetic processes you can represent the equation like in equation these equations are already derived and provided by several researchers and they are available in books. So for orthokinetic processes they can be written as  $\log\left(\frac{d_t}{d_o}\right) = A_2 + B_2 t$ , this  $d_o$  is the initial size of the particle,  $d_t$  is the enlarged size of the particle after time  $t$ . Okay, this  $A_1$ ,  $B_1$ ,  $A_2$ ,  $B_2$  are a kind of constants.

So these equations are valid for initial stages of agglomeration only, otherwise according to them if you keep on increasing the time then indefinitely increase the size of the particles, so that is, that we do not want. We want size enlargement but controlled size enlargement so that we can use them as a kind of workable units so that we can transport those solids comfortably and then subsequent process may also take place subsequently without any problem in flowability or conveying of those particles.

Okay, so that is the reason these equations are valid only for initial stages of agglomeration, otherwise they would indicate an indefinite increase of size  $d_t$  with time  $t$ .  $d_t$  is agglomerate size at time  $t$ . Dimensions of  $A_1$  is in  $m^3$ ,  $A_2$  is dimensionless,  $B_1$  is  $m^3/\text{sec}$  and then  $B_2$  is second inverse. These kind of units these constants are having.

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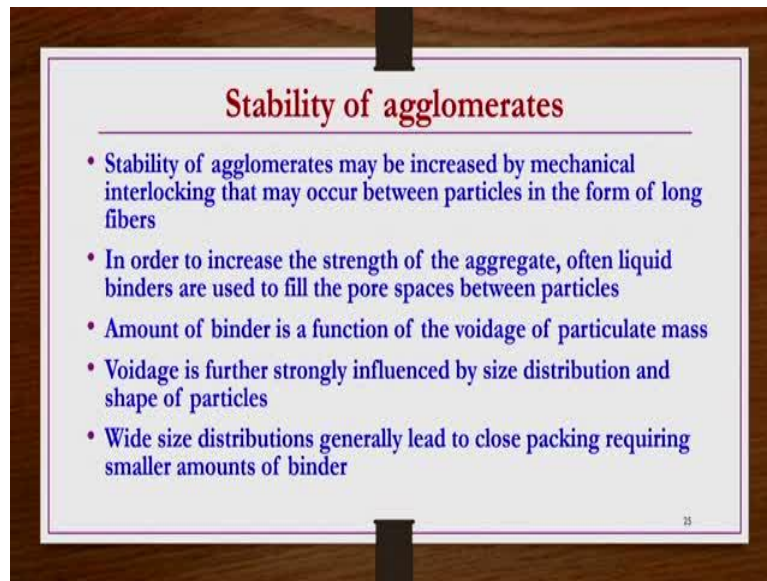


Then finally the characteristic time of a size enlargement process. There are different characteristic times are available but we take two of them which are famously accepted. Von Smoluchowski has characterised the suspensions by a ‘half-time’, half-time designated by  $t$  which is defined as the time taken to halve the number of original particles in a mono-disperse system. You have a mono-disperse system, let us say 1000 particles you have initially. So now you are doing agglomeration so that you know the number of particles are joining together and then bigger particles are forming. So, obviously number of particles will reduce though the weight may be remaining same.

So when these 1000 particles number of particles reduce to the 500 number of particles by agglomeration process, how much time it has taken, that is known as half-time and this time is taken as a kind of characteristic time by according to the researchers Von Smoluchowski. There is another characteristic time definition, Walton who defined an agglomerating system as one in which more than 10 percent of original number of particles has agglomerated in less than 1000 seconds.

You take a system, very fine particles let us say again you take you know 1000 number of very fine particles. So if 10 percent of those particles that is 100 particles if they are agglomerating within 1000 seconds, then you can say that system has a kind of agglomerating system according to Walton. These are the kind of few characteristic definitions, so which may be important in designing of you know these processes et cetera, size enlargement processes et cetera. They may be important in designing.

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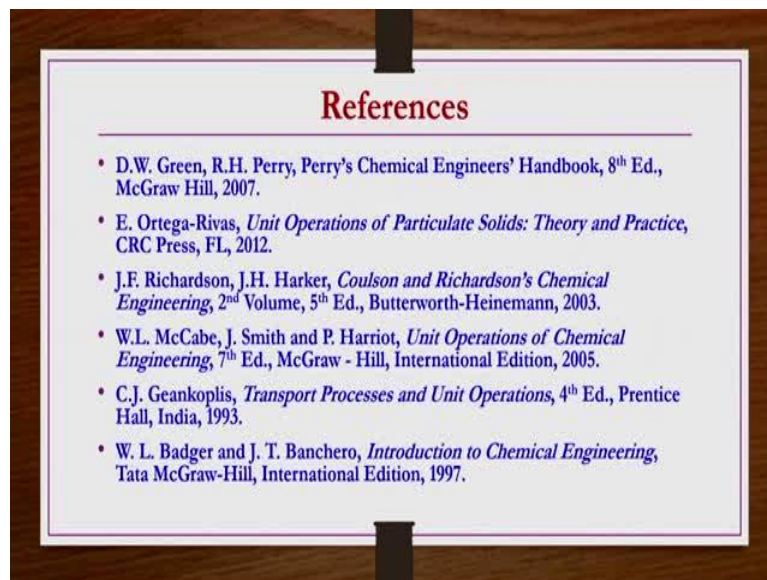
Stability of agglomerates may be increased by mechanical interlocking that may occur between particles in the form of long fibres. In order to increase the strength of the aggregate, often liquid binders are used to fill the pore spaces between particles. When you let us say when you take 3-4 particles, 2 particles like this another particle here, another particle here, so between the particles the interstitial spaces whatever are there those interstitial spaces may be filled by some kind of liquid binders so that the porous space between the particles that can be filled in and that after drying that will be providing a kind of enough strength for these segregates.

And then amount of binder is obviously function of the interstitial space of the particulate mass. Voidage or interstitial spaces that you can say so that how much binder, what is the amount of binder is required, obviously it will be a function of voidage of particulate mass or interstitial spaces of that particulate mass whatever we have taken. Voidage is further strongly influenced by the size distribution and shape of the particles. If the particles are very small finer sized particles, then voidage may be small, if you have a kind of a bigger particle or mixed particle kind of thing then voidage may be large.

So accordingly the shape of the particles you know if you have a spherical particles then voidage may be different. If you have the cylindrical particles then voidage may be different for a same packing height etc, for the same weight of the material, same materials, same density but the shape if you change then voidage, the voidage that is going to be formed because of these fine agglomerates is going to be different. That is what you mean by voidage is further strongly influenced by the size distribution and shape of particles. Wide size distributions generally lead to close packing requiring smaller amounts of binder.

So you know if you take the wider size distribution of the particles in general, then you need a less amount of binders for agglomeration because you know the voidage space is less or interstitial space is less in the case of wide size distribution. So that is the reason you know you may need only small amount of binder if you have a kind of wide size distribution particles. So this is, these are some details about the agglomeration, why we need the agglomeration, what are the agglomeration methods, how to do agglomeration by different types of forces et cetera and then typical agglomeration circuit et cetera that we have seen. We have also seen the agglomeration stability also we have seen. So next lecture we will be discussing about the equipment available for this size enlargement.

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The details presented in this lecture primarily are taken from this reference book, Perry's Chemical Engineers' Handbook by Green & Perry. Some information is also taken from Unit Operations of Particulate Solids: Theory and Practice, Ortega-Rivas. And then Coulson and Richardson's Chemical Engineering, 2<sup>nd</sup> Volume by Richardson and Harker. And then a few details from the Unit Operations of Chemical Engineering by McCabe, Smith and Harriot. Other references that may be useful, Transport Processes and Unit Operations by Geankoplis. And then Introduction to Chemical Engineering by Badger and Banchero. Thank you.