Fundamentals Of Particle And Fluid Solid Processing
Prof. Arnab Atta
Department of Chemical Engineering
Indian Institute of Technology, Kharagpur

Lecture – 48
Particle size enlargement (Contd.)

Hello, everyone and once again welcome back to the class of Fundamentals of Particle and Fluid Solid Processing. We I have started our discussion on the size enlargement process and we have seen the fundamentals that how this size enlargements are done one of such process is granulation.

So, in granulation there are several steps that we will be saying today and we started to understand that what are the forces that are required to have this granulation or are responsible for this granulation.

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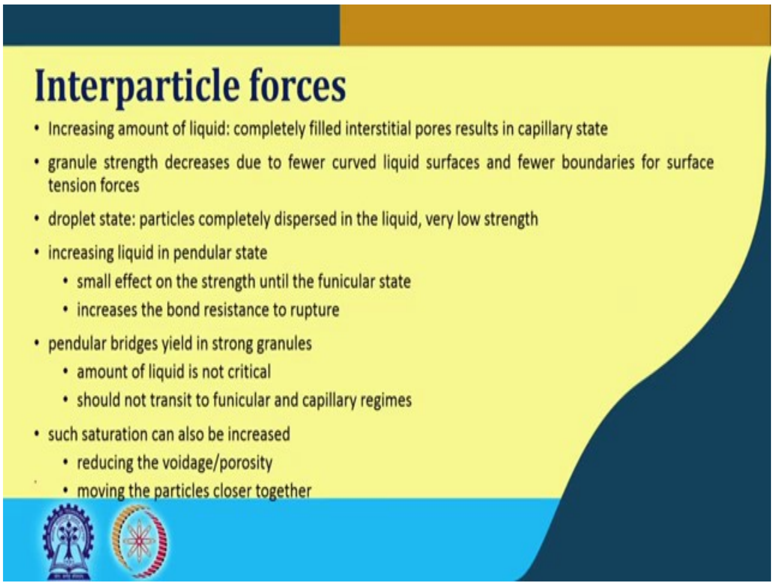

So, now we had come to this point in the last class that there are several interparticle forces and different state depending on the liquid saturation that we put on to the particle surfaces for the initial stage of granulation. So, we have seen four states that are where from the starting of granulation in the granulation state we have seen the funiculars state, pendular state, droplet state.

So, all these states what are what happens in those states we have briefly seen in the last class. So, we mentioned that this droplet state is actually when the particles are completely dispersed and we have seen the pendular state which is just the mere contact between the particles contact at the contact point we had a small amount of liquid was retained in that point.

So, as we increase the liquid amount there was a small effect on the strength until it becomes a funicular state and this amount of increase in liquid increases the bond resistance to rupture because what happens with this increased amount of liquid we can take these two particles away from each other without actually rupturing the bond because now the liquid saturations are higher or saturation is higher in that state. But, if it becomes funicular then this benefit of having pendular state is lost, because the benefits of pendular state is that it provides a strong granulation where the amount of liquid was not critical and, but we have to make sure that this should not go into the funicular of the capillary regime.

Now, such kind of saturation either we can increase the amount of liquid or we can compact the porosity or the voidage in between the particles. So, we can reduce the voidage or the porosity or we can move the particles closer together in order to increase the saturation; saturation is basically the volume of the liquid divided by the volume of the void space. So, as we decrease the void space the saturation increases.

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So, the other forces that are relevant in discussing the granular agglomeration is that the electrostatic forces. Now, this force is caused by the inter particle collisions and rubbing of particles against the equipment surface solid surfaces. So, it is the transfer of electron between the bodies and its force is basically proportional to the product of their charges. This force can be repulsive or attractive in nature and it does not require any contact between the particles and it is relatively long range force compared to the adhesional forces where the contact between the particles are necessary.

So, electrostatic force typically a long range force even compared to the van der Waals force, as well as the adhesional forces it is caused by the collision of different particles. Now, how the collision happens here because in granulation it is the agitation or the agitated movement of the equipment that induces movement in the particle also. So, this rubbing of the particle against each other as well as with the solid surfaces of the equipment, these electrostatic forces are generated.

Now, it is basically the transfer of energy transfer of electrons between the particles or the between the bodies its strength is proportional to the charges of the bodies; so, this force is also relevant when we discuss or when we think of the concept on the granulation.

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The other forces that are there is the solid bridges. Now, liquid bridges that we have discussed in the different structures of states of pendular, capillary, funicular and droplet states now the bridge that liquid bridges are not actually the ultimate stage for on the product generation.

Better permanent bonding can be achieved by these solid bridges than the liquid bridges. It has typically three forms, we see this kind of bridges in three different forms these are the crystalline bridges, liquid binder bridges and the solid binder bridges. So, say when the particles that will agglomerate to create this size enlargements, now if those particles are soluble in the binder or say it binder itself does not come itself it is typically comes in the form of a solution.

So, the particles are also soluble in that binder containing solution then what can be done that we can evaporate this whole thing and we can bring back the bridges between the particles by forming the crystalline structures after this liquid evaporation. So, basically the interstices liquid that has to be reduced the amount of liquid that are retain in between the particles are reduced now that is converted to the solid bridges by evaporation and crystallization. So, also it can be of like as I mentioned we initially give the binder, we provide the binder on the particle surfaces that and followed by evaporation we have this solid bridges.

Now, the other way can be introducing directly some solid binder now these solid binders are nothing, but very fine ground solids that can react with the liquid that is present in between the particles and form something the cement like structures and then it actually rigidly bonds the particles and the granulation happens the first granule can be formed in that way.

So, basically either if the particles are soluble in the solution containing binder, then after evaporation we can have the crystalline structures, or we can directly put the solid binders that actually adds reacts with the liquid that is present and it generates a cement like structure.

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So, if we relatively compare between the relative strength of this forces or when which one is dominant, we see that all interparticle forces are acting simultaneously. And, their relative importance depends on the particle property and the surrounding humidity change the operating atmosphere.

So, for example, for any aqueous system the absorbed moisture can considerably increase the van der Waal forces. If the surrounding atmosphere contains sufficient moisture then it can reduce the inter particle friction and the potential for interlocking which is not helpful. And, but it can help in improved powder flowability because these fine particles or we can say powders due to it is cohesive nature or adhesive nature sometimes it is difficult to flow, but the presence of humidity can improve it is flowability. But, the study has also been shown that if we increase the humidity to a greater extent where the powders exhibit this cohesive nature, then it can return to the cohesive kind of a state.

So, increasing humidity rapidly lowers the electrostatic forces and the powders that exhibit the shear say the cohesive in nature in dry atmosphere due to this electrostatic charging in between them, it becomes free flowing with increasing humidity. But again it can come back to its cohesive state if the humidity is sufficiently increased or enhanced to a greater extent the study or the research has shown this kind of properties as well. So, that means, there are several forces we have gone through now we have to understand it is relative importance, say, if we try to see in that in terms of the particle size.

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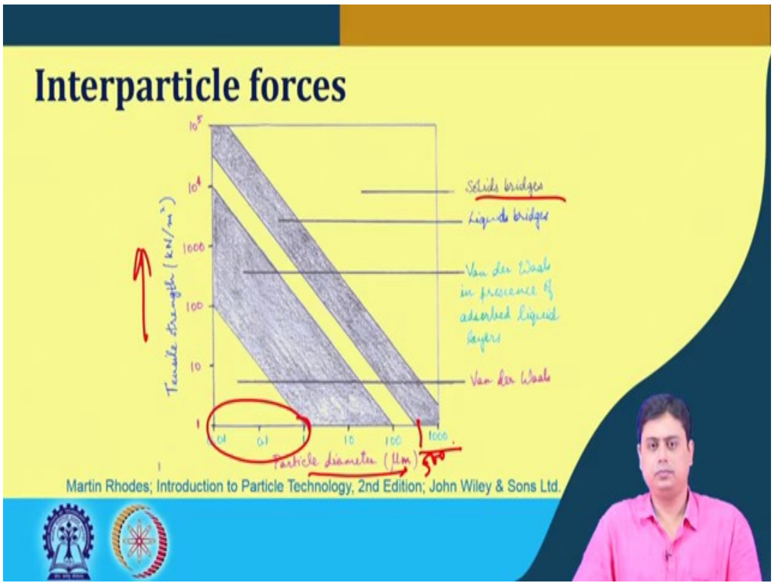

So, here in the x axis we have the particle diameter in micron and here the tensile strength for each of those forces.

Now, we can see this van der Waals quite logically is prominent or exist when the particle diameters are beyond 1 micron or below 1 micron specifically and the liquid bridges are there when the particle sizes are say beyond 500 µm. In between we have the van der Waal in presence of the absorbed liquid layer that you have seen. So, after liquid bridge if the particles are big enough then it can be held together by solid bridges otherwise the other forces are not dominant or not significant in that level.

So, if the particle size say goes beyond 100 µm or a 1000 µm. So, till say 1000 micron the liquid bridges can attract or can help in binding these two particles or multiple particles together, but beyond that size it actually requires solid bindings or solid bridges to bind the particles that will help in agglomeration.

So, this schematic basically summarizes the different interparticle forces that varies or it is importance with respect to the particle diameter the fine particle diameter which produces the agglomeration; the primary particles or the original particles from where the size enlargement we are trying to produce.

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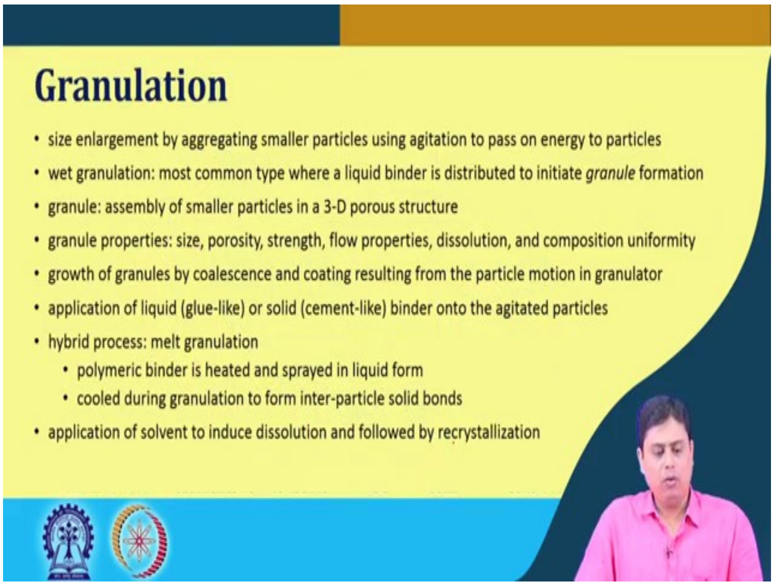

So, now if we particularly focus on granulation and look into the mechanism based on this force's the idea that we have got from this forces. We by now know that this size enlargement by granulation is basically aggregating smaller particles using agitation which pass on the energy or induces the energy to the particle. This agitation basically induces energy to the particle which helps in having aggregated granules.

Now, the most common method is the wet granulation; that means, where the liquid bounder is distributed over the surface of this fine powder or the particles to initiate the granule formation. Granule means it is the assembly of small particles in 3D porous structure. The intermediate space in between the particles we mentioned that is as voidage or the porosity.

So, wet granulation is most common and frequently used process where we spill some liquid binder on top of this particle surfaces to start the granulation process. Now, quite naturally this granule it is property depends on certain factor or the steps that are involved in granulation. Now, those properties are measured in terms of its size, porosity, strength, its flowability or flow properties its dissolution and composition uniformity. So, these are the parameters by which we understand the quality of granule.

Now, after distributing or spilling the liquid bounder on the top of the solid surfaces the granulation starts, say one granule has formed one cluster of particle has formed. Now, how it grows from a certain stage to its final stage that involves the coalitions or coating resulting from the particle movement in the granulator. So, as that granules now is moving due to the movement of the granulator, it sticks with the other particles fine particles say the individual particles or the primary particles it can be coated with those particles, it can coalesce with another granules and then this coalitions happens and its size enlargement continues.

So, there has been extensive application of liquid or solid like binder or say this liquid or solid binders, liquid kind as I mentioned glue kind of binders that helps to stick particles with each other, for solids it is a cement like binder in the agitated particles. Now, there can be hybrid process which involves or one of the process of this hybrid method is the melt granulation. So, where the thing happens is that if a binder is polymeric in nature or the polymer binder it is typically heated and sprayed in it the liquid form on top of the particles on the surface of the particles and it is cooled during the granulation to form interparticle solid bonds.

So, one hand we saw the formation of the solid bonds by crystallization. We saw the concentrated liquid bond by evaporating; we have also seen the introduction of solid binders to react with the solid particle the liquid that is entrapped in the porous zone to form a cement like structure or cement like bond. Now, in the hybrid process the solid the polymeric binder is initially generated in terms of a liquid form and then it is spread over the particles and then it is cooled during the granulation. So, simultaneous cooling happens when the granulation is happening and as it cools it forms a solid structure or solid bond in between the particles.

And, the other method that I have just mentioned is the application of solvent to induce dissolution to dissolve all the fine particles followed by recrystallization, so that we can have the crystal structure of the primary particles solids in nature.

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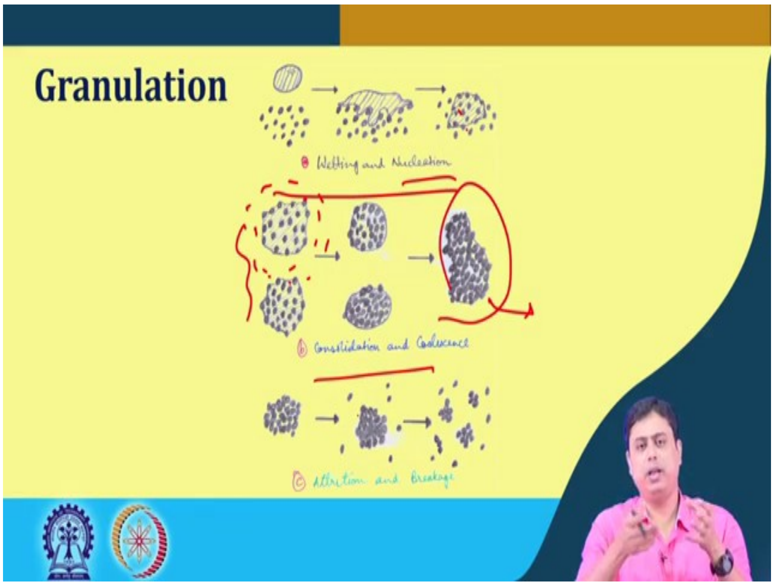

So, granulation basically involves three major rate process the rate of granulation is influenced by these rate processes one is the wetting and nucleation. The first step is the wetting and nucleation, the second step is the consolidation plus the coalitions; so, consolidation and coalitions and the last step is the attrition and breakage. So, what happens, say, we have this fine particles assembly of fine particles collection of fine particles. A liquid is dropped on the surface it should wet the particle surfaces; it should penetrate the depth of that powder bed. So, the wetting is what important as the first place.

Now, this wetting starts the formation of granulation that is called the nucleation. Now, once one granule is formed then the coalitions between two granules or say coating with the finer particles further due to it is movement produces a bigger structure or the consolidation of granules and then further the movement of these granules are there in the granulator. So, as the movement continues the this granulation movement is there so, it undergoes attrition not only coalitions happens it also goes for coalitions, attritions happens say with the solid surfaces of the equipment, it followed by the breakage of certain bigger sized that cannot withstand the bond strength.

So, if this attrition level goes beyond this bond strength values, then definitely that particle will again break or some of the particle will come out from that bigger granules because these bridges that we have formed. Now, these bridges either can be formed in different ways that we have just discussed, either by liquid bridges or by solid bridges. If it is solid bridge then a bigger size of the granule can be retained during the attrition as well, but if it is of liquid bridge and the attrition is happening in that state itself before it is freezed to a solid state then that particle that granule will decay in size due to this attrition the breakage will happen.

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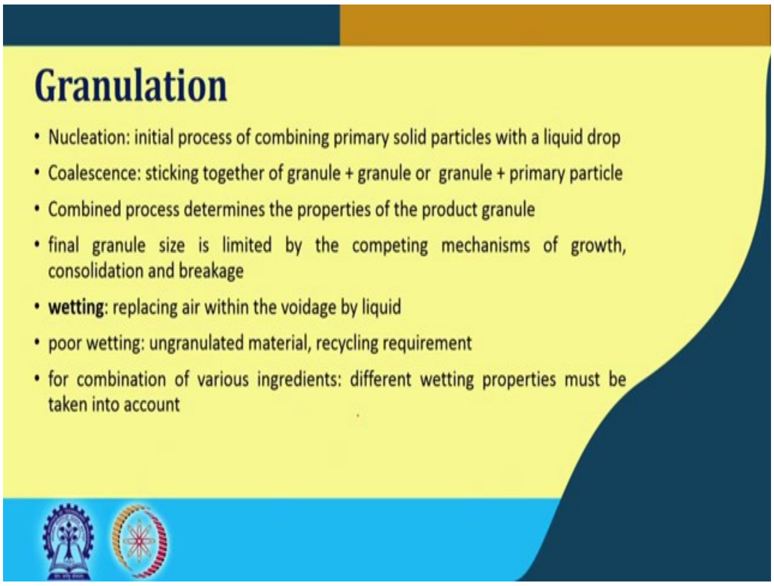

So, which means we have nucleation at the first place which is the initial process of combining primary solid particles with a liquid drop. It is followed by coalescence: coalescence means sticking together the granules either with each other or with the primary particles; so, basically coalescence and coating or combination of these two. Now, this combined process determines the property of the product granule that the way it is being formed that dictates that what would be the porosity inside those granules, what is the orientation of the particles, the uniformity inside the structure and these basically dictates the granule size and its properties.

Now, the ultimate the end product its granule size is limited or restricted by the competing mechanism of this growth, consolidation and breakage. These three rate steps that are there in granulation. So, firstly, it is wetting plus nucleation followed by coalitions and consolidation or its growth and final step is the breakage for due to attrition. So, these three steps and its competing mechanism dictates the final size of the end granule or the end product.

So, which means if we now go into the details of the stay which is the wetting plus nucleation we see that wetting is basically replacing the void space that is filled with air by some liquid or filling off that void with the liquid. Now, this wetting rate has tremendous influence on this granulation rate because if there is poor wetting then the particles will not be bridged together.

So, which means there will be ungranulated material; that means, the materials have to be recycled again as feed for the granulation for the wetting to happen and say in this granulation or sized enlargement. If you are mixing several different ingredients that typically happens in pharmaceutical industry; different drug elements say are mixed to form a tablet. Now, if those are of different ingredients; that means, of different material which means those materials will have their own wetting property.

So, during this step different wetting properties must be taken into account different weighting properties means different weighting properties due to different material present during this combination. So, we will see in detail that how the weighting rate influences this granulation or how it is determined how researchers are explored this area and we will see followed by how the coalitions happens and we can understand the breakage phenomena.

So, in the next class, we will be going into the details of this wetting phenomena and the nucleation step focusing mainly on the granulation. Until then, thank you for your attention.