Fundamentals Of Particle And Fluid Solid Processing
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Lecture - 50
Particle size enlargement (Contd.)

Hello everyone and welcome back once again in the class of Fundamentals of Particle and Fluid Solid Processing. And we were discussing about Particle size enlargement. We have seen mainly the granulation through which we enlarge the particle size.

Now, in granulations there are different stages that we have seen, there are different rate determining processes. We started with the wetting and nucleation; we have seen a bit of the collision and the coalescence followed by the consolidation of these granules. And there is the breakage or the fragmentation and the attrition of this part. So, these three categories, this three types of this processes basically determines the granulation, the rate of granulation or the degree of granulation.

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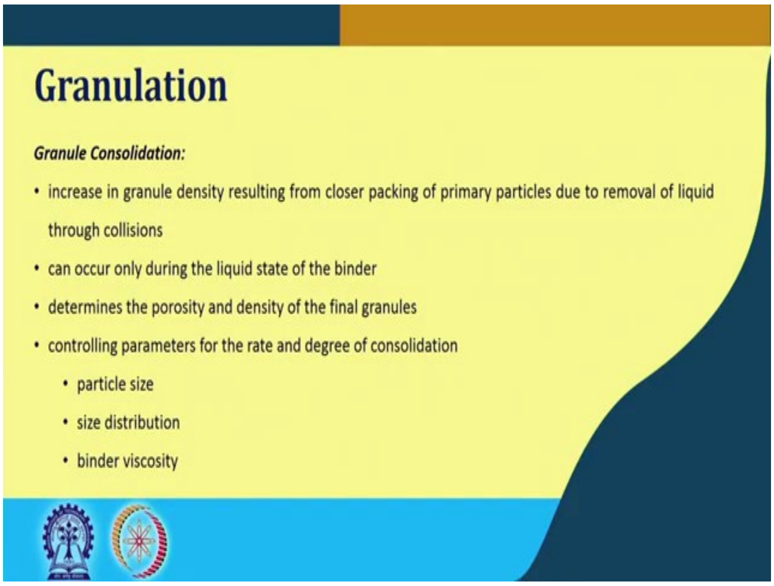

Granulation

Granule Consolidation:

- increase in granule density resulting from closer packing of primary particles due to removal of liquid through collisions
- can occur only during the liquid state of the binder
- determines the porosity and density of the final granules
- controlling parameters for the rate and degree of consolidation
  - particle size
  - size distribution
  - binder viscosity

Now, we have seen this granule consolidation stays this slide in the last class which shows that this consolidation is the increase in granule density resulting from closer packing of primary particles due to removal of liquid through collisions. Now, this consolidation can occur only when or until the stage when the binder or the (Refer Time: 02:06) that are binding that particles are in liquid state. And this consolidation step also determines the porosity and density of the final granules. The controlling parameter for this rate and degree of consolidation are particle size its distribution and the binder viscosity.

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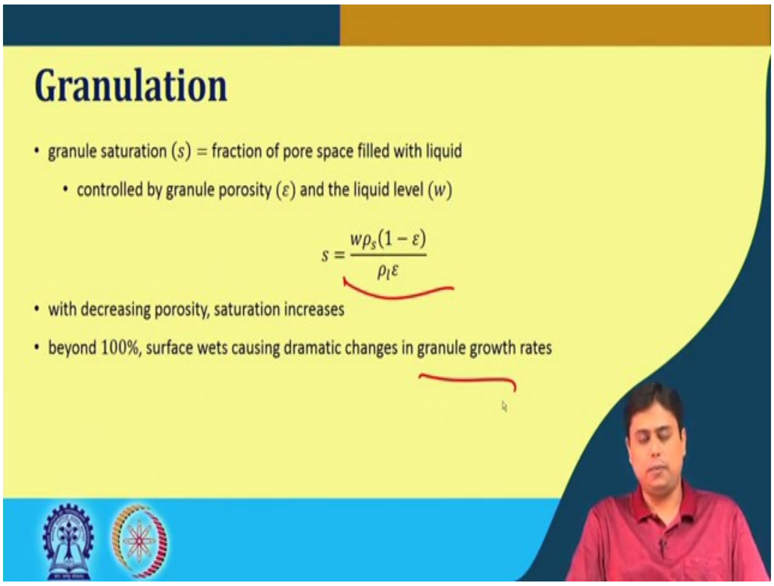

Granulation

- granule saturation (s) = fraction of pore space filled with liquid
  - controlled by granule porosity (ε) and the liquid level (w)

$$s = \frac{w\rho_s(1-\varepsilon)}{\rho_l \varepsilon}$$

- with decreasing porosity, saturation increases
- beyond 100%, surface wets causing dramatic changes in granule growth rates

Now, this granular saturation which can be defined as a fraction of pore space filled by liquid which is controlled by the granule porosity and the liquid level. So, with decreasing porosity from this expression that is given here

$$s = \frac{w\rho_s(1-\varepsilon)}{\rho_l \varepsilon}$$

we can understand that the saturation increases. And beyond 100% saturation there is liquids come out from the interstates this it causes the surface wetting which has the dramatic effect on the granule growth and its rate.

Now, we will now look into the details of this granule growth rate. This is the part we have seen what the weighting of this phenomena.

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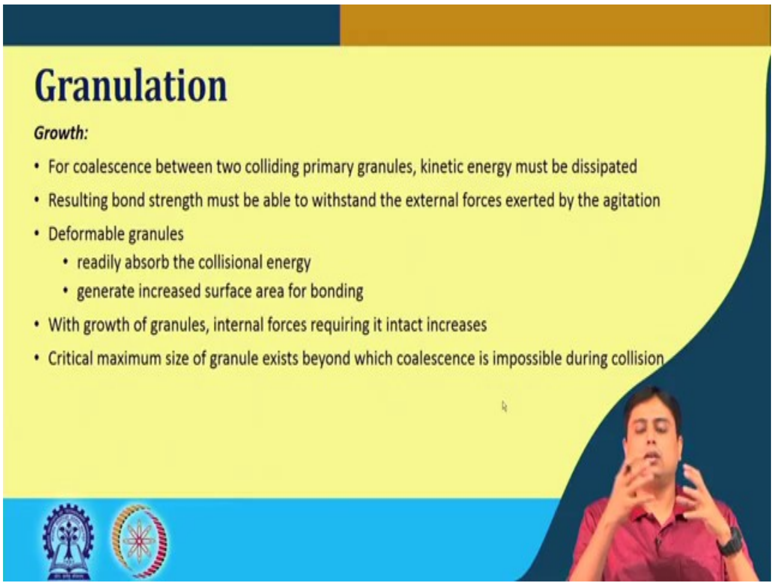

Granulation

Growth:

- For coalescence between two colliding primary granules, kinetic energy must be dissipated
- Resulting bond strength must be able to withstand the external forces exerted by the agitation
- Deformable granules
  - readily absorb the collisional energy
  - generate increased surface area for bonding
- With growth of granules, internal forces requiring it intact increases
- Critical maximum size of granule exists beyond which coalescence is impossible during collision

Now, in growth state or the growth stage of granulation, it is nothing but the coalescence between two colliding primary granules and then for this the kinetic energy of this collision must be dissipated. And the resulting bond strength it must be able to withstand the external force that is impacted by the agitation of the granulators.

So, two granules are colliding and it is coalescing. Now, it forms a bigger granule. Now this bigger one should be able to withstand the external force that is imparted by the agitation of the granulator. So, the bond strength should be sufficient enough. Now, in case of deformable granules; that means, if the surfaces can be deformed during the collision it can readily absorbed the collision energy and it generate the increased surface area for the bonding to happen.

Now with the growth of this granule, internal force also the it requires more and more internal force to make intact and because this internet force requirement also increases with the growth. As I said that as the granules become bigger and bigger, so the internal force that it requires to keep it intact or keep it more stabled, that force requirement increases. Now there is a possibility and the researchers have shown that there is a critical maximum size of this granule that exists beyond which the coalescence is impossible during the collision. So, the possibility of collisions; that means, not all collisions are resulting in coalescence.

So, there is a maximum possible size because this is a essentially the interplay between this internal bond strength and the collisional energy. So, not all the collisions will create coalescence or we will have coalescence.

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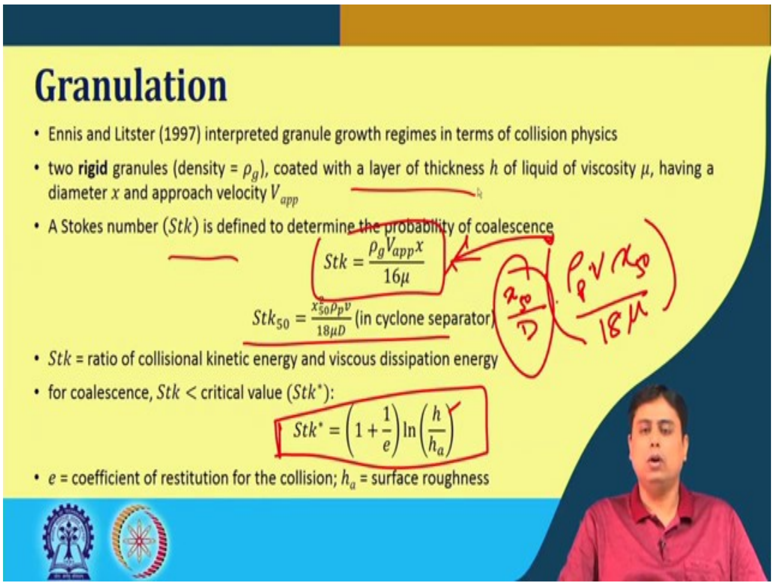

Granulation

- Ennis and Litster (1997) interpreted granule growth regimes in terms of collision physics
- two **rigid** granules (density = ρ_g), coated with a layer of thickness h of liquid of viscosity μ, having a diameter x and approach velocity V_{app}
- A Stokes number (Stk) is defined to determine the probability of coalescence

$$\boxed{Stk = \frac{\rho_g V_{app} x}{16\mu}}$$

$$Stk_{50} = \frac{x_{50}^2 \rho_p v}{18\mu D} \text{ (in cyclone separator)}$$

$$\frac{x_{50}}{D}\left(\frac{\rho_p v x_{50}}{18\mu}\right)$$

- Stk = ratio of collisional kinetic energy and viscous dissipation energy
- for coalescence, $Stk <$ critical value (Stk^*):

$$\boxed{Stk^* = \left(1 + \frac{1}{e}\right) \ln\left(\frac{h}{h_a}\right)}$$

- e = coefficient of restitution for the collision; h_a = surface roughness

So, researchers have shown this granule growth regime in terms of collision physics; say to rigid bodies or two rigid granules. Now region means there are not they are not deformable. So, 2 hard granules of density rho g coated with a layer of thickness h of the liquid that has a viscosity mu having a diameter x and approach velocity of a V_{app}. Now, the possibility of coalescence has been determined based on a stoke number.

$$Stk = \frac{\rho_g V_{app} x}{16\mu}$$

Now, this stoke number that is presented here to dictate the possibility of coalescence converting to coalescence is different from what we have seen in cyclone separator the Stokes number.

$$Stk_{50} = \frac{x_{50}^2 \rho_p v}{18\mu D}$$

That is there the Stokes number was the measure of efficiency, it relates the particle diameter with the cyclone diameter. So, basically this component is analogous, but this is the particle diameter and the cyclone diameter was incorporated.

So, this Stokes number, the senses it is different from here what is defined as the Stokes number. Now here the Stokes number is the ratio of collisional kinetic energy and the viscous dissipation energy or the energy that dissipates by viscous dissipation. So, in order to happen this coalescences this Stokes number has to be a lesser than a critical value that is say the critical Stokes number. And it is defined as this expression,

$$Stk^{\text{¿}} = \left(1+\frac{1}{e}\right)\ln\left(\frac{h}{h_a}\right)$$

where e is the coefficient of restitution for the collision and h_a is the surface roughness and h I already mentioned the layer of coating thickness of the liquid.

So, if this Stokes number becomes a lesser then the critical value then all collision; all collisions will be converted to coalescence.

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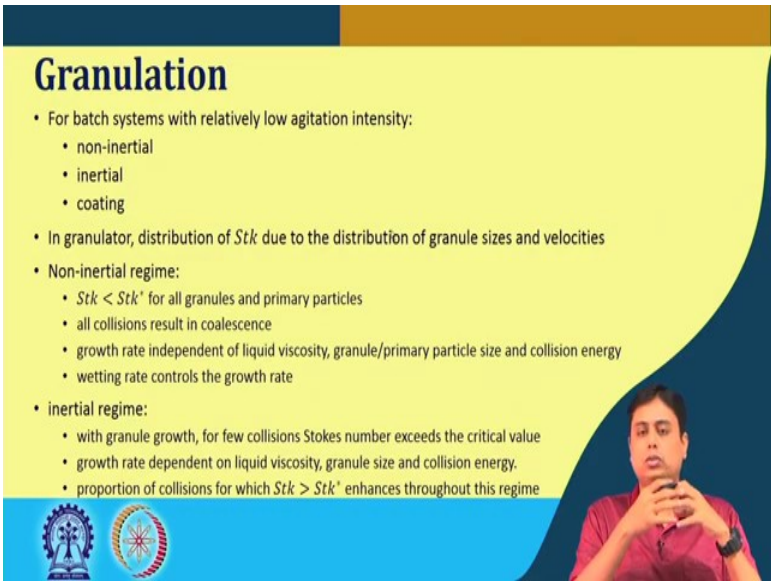

Granulation

- For batch systems with relatively low agitation intensity:
  - non-inertial
  - inertial
  - coating
- In granulator, distribution of Stk due to the distribution of granule sizes and velocities
- Non-inertial regime:
  - $Stk < Stk^*$ for all granules and primary particles
  - all collisions result in coalescence
  - growth rate independent of liquid viscosity, granule/primary particle size and collision energy
  - wetting rate controls the growth rate
- inertial regime:
  - with granule growth, for few collisions Stokes number exceeds the critical value
  - growth rate dependent on liquid viscosity, granule size and collision energy.
  - proportion of collisions for which $Stk > Stk^*$ enhances throughout this regime

Now, based on this hypothesis for a batch system with relatively low agitation intensity 3 different regimes have been identified; one is non inertial, inertial and the coating regimes. Now, in granulator due to the distribution of granule sizes and its velocities as well as the primary particle size variations, there is a distribution of Stokes number. In non inertial regime this Stokes number are always lesser than the critical Stokes number, for all the k all the particles including the granules.

So, all collisions basically converted to coalescence and the growth rate and then quite naturally is independent of the liquid viscosity granule or the primary particle size and the collision energy, this wetting rate actually what controls the growth rate. And that is why in the last slides it is mentioned that wetting the saturation when it goes beyond 100 percent when the liquid comes up on the surfaces it drastically changes the growth mechanism.

On the contrary, this inertial regime as the granules growth a few collisions of these granules for which the Stokes number exceeds the critical value or the critical Stokes number, because if we look at the expression we see the Stokes number is dependent on the diameter is directly proportional to the diameter. So, as the granule goes in size the Stokes number also increases.

And in few cases if it increases then; that means, those collisions even if it happens will not be converted to coalescence or the growth of the granules. So, the growth rate in this regime depends on the liquid viscosity, granule size and the collisional energy. The proportional of collisions which exceeds this for which the Stokes number exceeds this critical Stokes number, it throughout increases in this regime.

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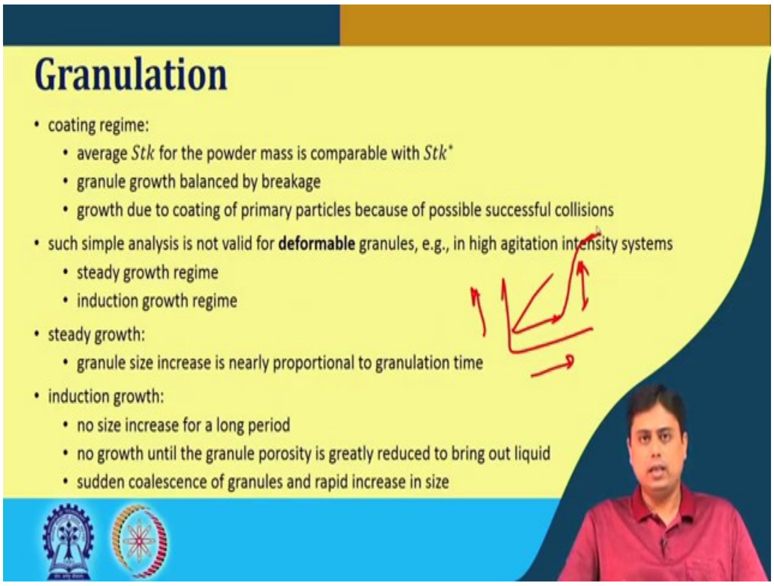

Granulation

- coating regime:
  - average *Stk* for the powder mass is comparable with *Stk**
  - granule growth balanced by breakage
  - growth due to coating of primary particles because of possible successful collisions
- such simple analysis is not valid for **deformable** granules, e.g., in high agitation intensity systems
  - steady growth regime
  - induction growth regime
- steady growth:
  - granule size increase is nearly proportional to granulation time
- induction growth:
  - no size increase for a long period
  - no growth until the granule porosity is greatly reduced to bring out liquid
  - sudden coalescence of granules and rapid increase in size

And when this average Stokes number by mass of this full powder is comparable with the critical Stokes number then the granule growth is balanced by the breakage.

And here in this stage is called the coating regime because the growth is now mainly due to the coating of primary particles with this grown granules because in these cases the there are fine particles fine primary particles for which there is possibility of having Stokes number lesser than the critical Stokes number. So, by this hypothesis still there are chances that when those primal particles hits the granules it sticks to the surface or the coating happens.

Now, this simple analysis does not work if the granules are deformable and that typically happens in high agitation intensity system. In that case two different regimes have been identified one is the steady growth regime the other is the induction growth regime.

So, in steady growth regime what happens, the granule size increases nearly proportional to the granulation time, which means if you draw a graph of granulation time and granule growth its size, it linearly increases. In case of induction growth for the duration there is no size change or no increase in sizes, and this no growth regime exist till a period until and unless this liquid that is there in the voidage comes out on the surface. As it comes out of the surface; that means, somehow if you can reduce the granule porosity to a greater extent, this liquid will be squeezed out and then that comes onto the surface of the particle.

And this is the kind of scenario that is also this beyond 100 percent saturation and in that case a sudden coalescence of the granules will happen and rapid increase in size will be there. So, in this case if you draw this graph there will be a no size change period and then there will be a sudden jump in the particle size growth. So, that this is the induction growth regime, in case of deformable granules.

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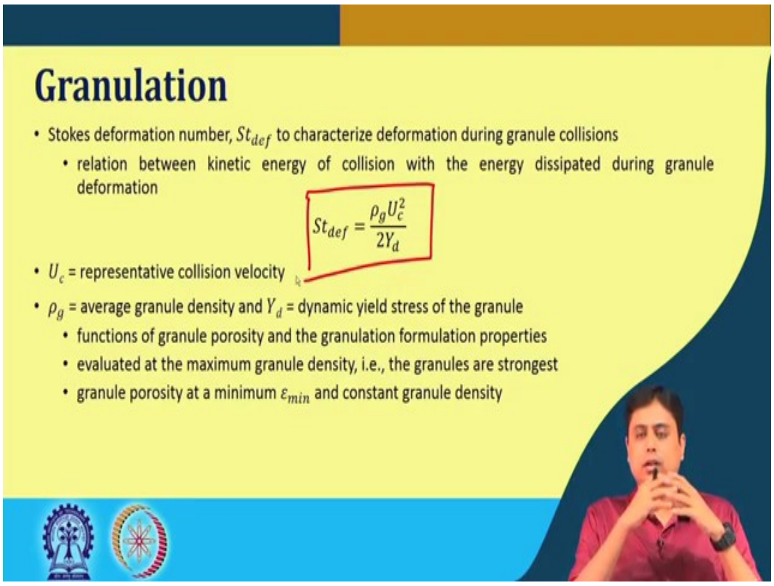

Granulation

- Stokes deformation number, St_{def} to characterize deformation during granule collisions
  - relation between kinetic energy of collision with the energy dissipated during granule deformation

$$\boxed{St_{def} = \frac{\rho_g U_c^2}{2Y_d}}$$

- U_c = representative collision velocity
- ρ_g = average granule density and Y_d = dynamic yield stress of the granule
  - functions of granule porosity and the granulation formulation properties
  - evaluated at the maximum granule density, i.e., the granules are strongest
  - granule porosity at a minimum ε_{min} and constant granule density

Now, this deformation during this granulation, in order to characterize this Stokes deformation number has been defined which relates the kinetic energy of collision to the energy dissipated during granule deformation.

So, this Stokes number here, now Stokes deformation number is defined in this manner,

$$St_{¿}=\frac{\rho_g U_c^2}{2Y_d}$$

where Uc is the representative collisional velocity ρ_g and Y_d are the average granule density and dynamic yield stress of the granule respectively. Now, these are strong functions of the granule porosity and granulation formation properties which are typically evaluated at the maximum granule density which means when the granules are in the strongest form and this happens when the granule porosity is at its minimum and the granule density does not change further or it does not alter after that period because that is the minimum voidage it can achieve it has become such concentrated granule.

So Stokes deformation number help us to identify the deformation during the granule collision. It relates the kinetic energy of collision with the energy dissipated during the granule deformation.

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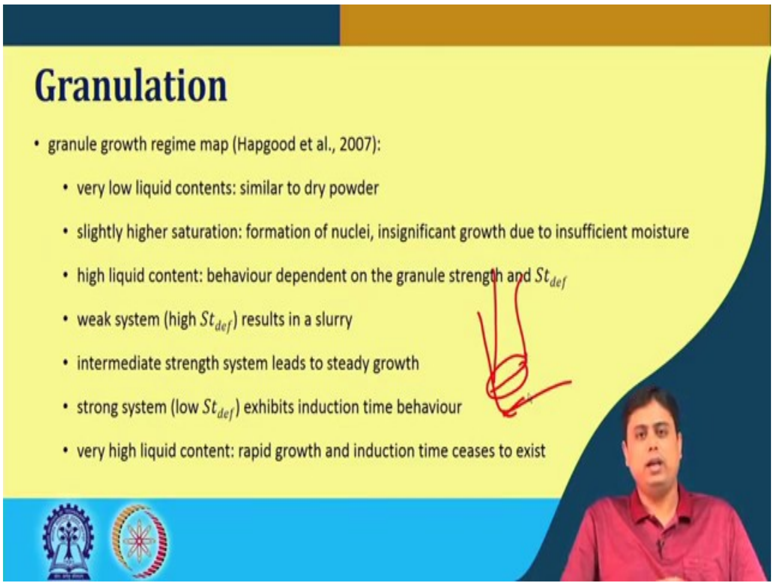

Granulation

- granule growth regime map (Hapgood et al., 2007):
  - very low liquid contents: similar to dry powder
  - slightly higher saturation: formation of nuclei, insignificant growth due to insufficient moisture
  - high liquid content: behaviour dependent on the granule strength and St_{def}
  - weak system (high St_{def}) results in a slurry
  - intermediate strength system leads to steady growth
  - strong system (low St_{def}) exhibits induction time behaviour
  - very high liquid content: rapid growth and induction time ceases to exist

So, if these are combined then we can have a granule growth rate regime map. Now, in this regime map we see that had very low liquid content, the product size that product state is basically similar to that of a dry powder, which is difficult to flow.

So, if there is a very low liquid content the solvent is very low in amount we can have whatever we started with that kind of a condition. We increase this liquid contents slightly; that means, we increase it to a higher saturation it starts to form the nucleus. So, several granules or nuclei form, but their growth is insufficient because of the in sufficient amount of moisture present or the liquid present.

So, if we increase the liquid content further say at a high liquid content then the behaviour depends on the granular strength and this Stokes deformation number. So, at a moderately higher liquid content the product granule size or the its behavior is dependent on the granule strength and the Stokes deformation number. So, for weak a system which means high stoke deformation number results in a slurry. The intermediate strength system leads to steady growth, at load deformation number or Stokes deformation number; that means, when the system is very strong it exhibits the induction growth behaviour or induction time behaviour.

If we further increase this liquid content to a very high level a say the extremely high liquid content then that rapid growth will be observed or has been observed and it has been seen that this induction time approaches 0; which means the graph would then look like a very rapid increase in the growth, instead of having any induction time and then its growth.

So, as the saturation increases this period goes lower and lower. So, with such detailed study a flow regime map of this kind has been developed and it has been verified with the several materials, several types of powders and it has been seen that in granulation these stages indeed happen.

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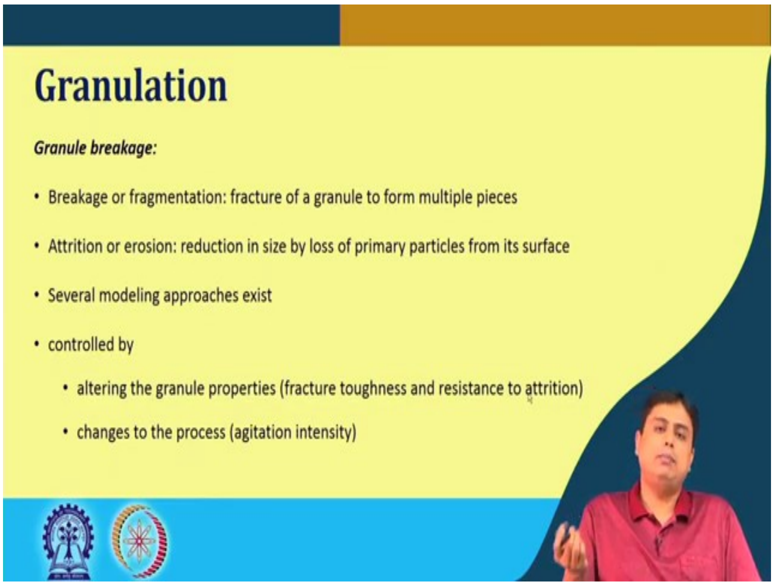

Granulation

Granule breakage:

- Breakage or fragmentation: fracture of a granule to form multiple pieces
- Attrition or erosion: reduction in size by loss of primary particles from its surface
- Several modeling approaches exist
- controlled by
  - altering the granule properties (fracture toughness and resistance to attrition)
  - changes to the process (agitation intensity)

And then it is followed by the granule breakage, the attrition and the breakage. So, the breakage or the fragmentation is the fracture of granules into multiple particles or multiple pieces, on the other hand attrition which is also called erosion is the reduction in size by the loss of primary particles from its surface.

So, both the things happens at a certain point or after the growth stage, after the granule has reached its sufficient growth stage depending on the granule strength on the internal bond strength this breakage will then followed by. There are several modeling approaches exist to explain the mechanism of this breakage which are mainly controlled this breakage mechanism is mainly controlled, can be controlled rather by altering the granule property or by changing the process operation.

So, by changing the granule property means say either you can increase the toughness of the material in order to resist in the fracture or to resist the attrition. So, the breakage can be reduced or by change in process operation means for example, you can change the agitation intensity of the granulator say you operate at a reduced RPM, in that way you can reduce the breakage amount.

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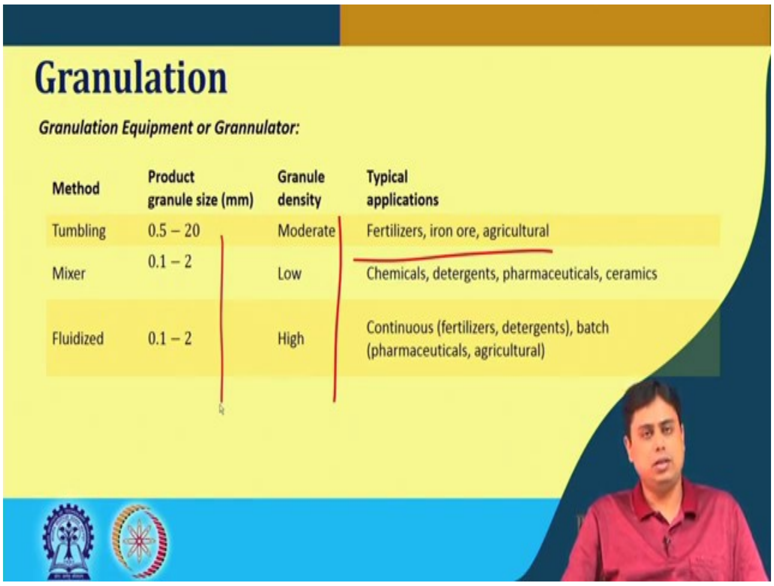

Granulation

Granulation Equipment or Grannulator:

Method	Product granule size (mm)	Granule density	Typical applications
Tumbling	0.5 – 20	Moderate	Fertilizers, iron ore, agricultural
Mixer	0.1 – 2	Low	Chemicals, detergents, pharmaceuticals, ceramics
Fluidized	0.1 – 2	High	Continuous (fertilizers, detergents), batch (pharmaceuticals, agricultural)

So, these are the steps in the granulation that we have gone in details. Now we will quickly look to the overview of couple of granulators.

Now, this granulators there are most commonly used can be categorization in these three forms the tumbling, granulator mixer, granulator and fluidized bed granulator. Depending on the granular density this is also can be categorized that the tumbling and granulator can handle moderately a moderately dense granules or it generates that kind of end products. Mixer granules has low granule density, the fluidized granulators has a very high granule density. These are used in these respective areas, the tumbling granulators are typically used in fertilizers, iron ore, processing in agricultural industries.

Mixer granulators are frequently use in detergent or the pharmaceutical industry, fluidized bed typically used in both continuous and batch operations in, and it has possibly the most extensive use. The product size, the product granule size are also listed here that is the tumbling granulator typically the product granule size is from 0.5 mm to 20 mm fluidized bed and the mixer granulators liters typically results in 0.1 to 2 mm size granules.

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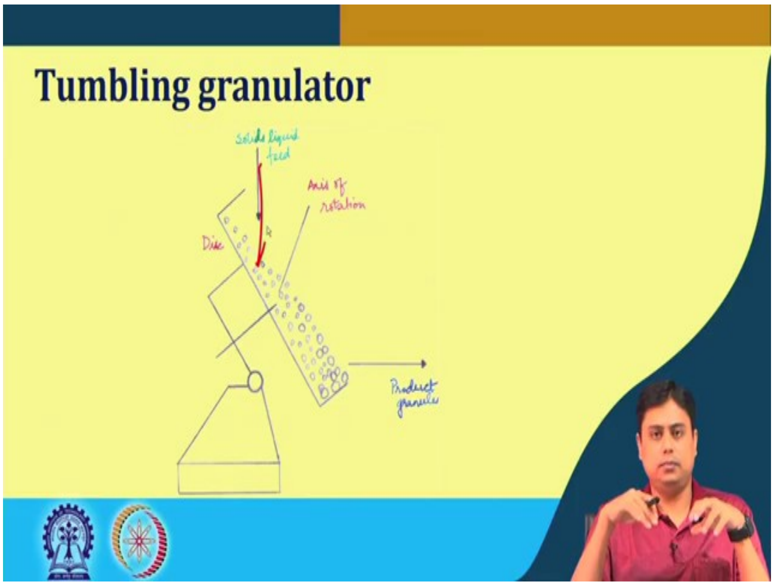

Tumbling granulator

Tumbling granulator a schematic of that this is a disc granulator this has a tumbling motion the disc. So, this tumbling motion creates this granulation or results in granulation and as well as due to this tumbling motion of this disc there is a classification happens in the product. So, the product size typically has a narrow size distribution. So, here all the things are fed together, that the liquid, and the solid mixture of the solid and the liquid binders. It rotates as well as it tumbles and then it creates the product of narrow size distribution.

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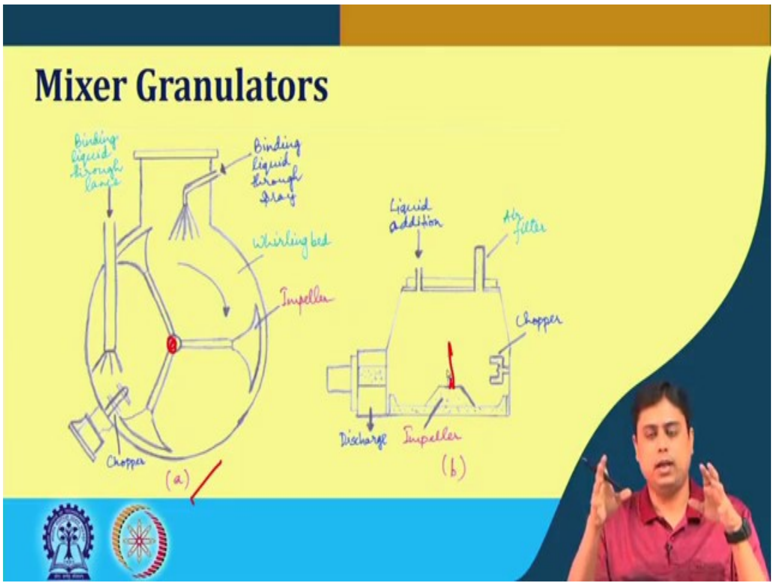

Mixer Granulators

In mixer granulator it can be of two types, one is the horizontal axis the other is the, this is the horizontal axis and this is the vertical axis. So, here the impellers are there, it works on the concept of mixing and chopping the binders and the liquids and the solid particles are fed in. It gives the motion by the impeller; the granulation happens this can be operated in a range of RPMs from low to very high. So, low say in the range of a 100 RPM to 3000 RPMs as high as possible.

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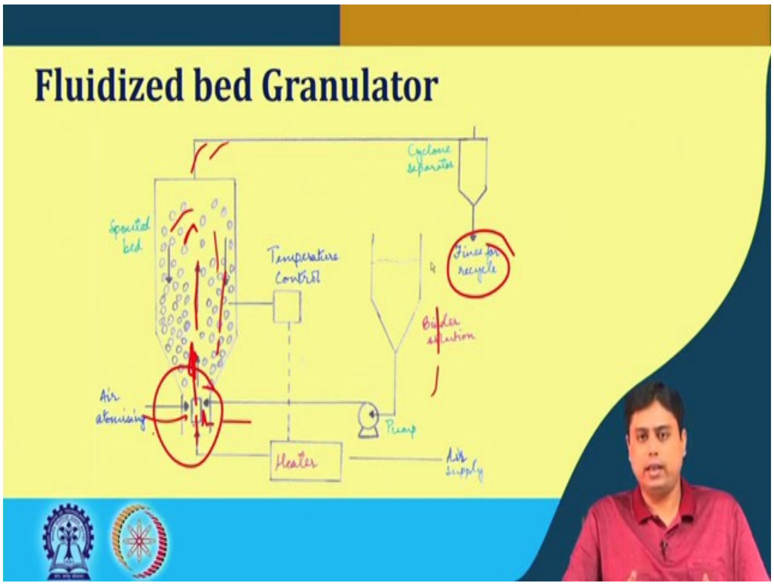

Fluidized bed Granulator

In case of fluidized bed granulator this is the classical fluidized bed that we have seen. So, this is here this is a spouted bed where this binders can be fed from the top or within this fluidized bed. So, here say the binder solution comes from here it is pumped also there is the atomizer which sprays the binder solutions into the fine particles and then we can see there will be granulation inside the bed. We can control the temperature and then we can have the finer particles for recycle.

Now in this case finer particles are not the desired product, we are doing size enlargement of the finer particles. So, the desired size particles will be collected at the bottom and the finer size particles will be collected at the top which will again go to the recycle.

So the reason I mentioned that this is possibly the extensive granulator that has find application is because the temperature control in this granulator, the transfer of energy everything is efficient than the others. Because the whatever the advantage we had for fluidized bed it is here and also we have seen that it can be operated in batch mode or in continuous mode operation. So, it gives the flexibility of operation as well. So, what we have seen throughout the size the enlargement starting from its utility why it is required, its processes and particularly one process that is the granulation. We had different stages in granulation, the different rate processes in granulation we have seen in details.

We have briefly gone through the types of granulators, and its working principle for detailed this of these granulators and its designed and the other granulators one can always refer to Perry’s handbook or the encyclopedia by (Refer Time: 29:25). With this I conclude the size enlargement section here and on the next day we will see with the other section.

Thank you for your attention.