

**Fundamentals Of Particle And Fluid Solid Processing**  
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**Lecture - 47**  
**Particle Size enlargement**

Hello everyone and welcome back to the class of Fundamentals of Particle and Fluid Solid Processing. Today we will start our discussion on Particle Size enlargement; we have seen a relatively detailed discussion on the particle size reduction. Now, we will see that why enlargement is also important.

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**Particle size enlargement**

- Formation of larger masses from smaller particles keeping the identity of original particles
- Vital step in particulate process industries
- Extensive application in pharmaceutical, agricultural and food industries, and also in minerals, metallurgical and ceramics industries
- Reduction of
  - dust hazard
  - caking and lump formation
- Improvement of flow properties
- Increase in bulk density for storage
- Controlled use of active ingredient
- Control over surface to volume ratio

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So, size enlargement by its name you can understand it is the formation of larger masses from smaller particles, but keeping this identity of the original particles intact. So, it is basically a formation of agglomeration say it is a vital step in various process industries.

And particularly it finds extensive application in pharmaceutical, agriculture, and food industry, it is also used in several other industries as well like in minerals, metallurgy and ceramic industries. So, where basically we have already finer particles or fine particles, but we need some agglomerates and why is so, because by doing so, we can reduce the hazards that is associated with this fine particles for example, the dust hazard.

It is safety hazard or health hazard related to the fine particles, because one of the most detrimental dusts for our human respiration is the asbestos. Now it is the size distribution is the size particularly that is detrimental for our lungs. So, it is the finer size of the asbestos that is actually a very very dangerous for our health. It also reduces the chances when when we make the these smaller particles together we combined them we bind them. It also reduces this caking and lump formation tendency when these are together.

So, basically say some fine particles you have a bulk of fine particles that you intend to flow, it is not easy to make them flow, instead if you had some aggregates of these smaller particles it can roll over very quickly. So, which means by enlarging it is size we also improve it is flowability or the flow property. We increase the bulk density for it storage, also by increasing this size of a particles in terms of aggregates or granules. We control the use of active ingredients in there which is very much helpful in the drug industries or the pharmaceutical industries.

And also when we have a reasonably bigger size than the ultrafine particle also the fine particles it helps us to have a control over the surface to volume ratio or the specific surface area. Now, the importance of these we have already seen in the last classes. So, not only the size reduction is important size enlargement is equally important in several industries and the foremost reason one of the foremost reasons is the reduction of dust hazard or the health hazard with the fine particles. The second most important I would say is the improvement of it is flowability. So, all these reasons leads to discussing this topic that why size enlargement and how it is done.

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**Granulation**

- Agglomeration: formation of agglomerates or aggregates smaller particles
- Granulation: agglomeration by agitation methods
- Types of interparticle forces and their relative importance
- Steps in granulation: wetting, growth, consolidation and attrition
- Brief overview of industrial granulation equipment

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So, agglomeration that I am mentioning is the formation of agglomerates or say the aggregates of smaller particles. Now, granulation is a strategy of this agglomeration by agitation methods. There are several methods of enlarging sizes like the granulation, drilling operations, there are other operations we will mention in due time.

But let us focus primarily here on the granulation, because of it is very common use or extensive use like we have only chosen ball mill in the case of size reducing equipment and has seen it is details. Similarly, here we will take this granulation operation as one of the size enlargement process although there are several processes. We will focus on this one mostly in this section and we will see that, what are the forces at the molecular level that is involved in this granulation process? For what are the forces that are dominant that this granulation process during this what are the forces we have.

So, for this what we will see, we will see the types of inter particle forces and their relative importance in the granulation process. There are different steps in this overall granulation process like the wetting, growth, consolidation, attrition these are the steps we will see in our brief details and we will go in the brief overview of the industrial granulation equipment to complete our section.

So, which means again as a size enlargement process we will mostly focus on granulation which is agglomeration by agitation, we will see it is various stages, we will look into the various molecular level forces that are important in having this granulation efficiently and

then at last we will have an overview of various granulation equipment that we did like in case of size reduction.

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**Interparticle forces**

**van der Waals Forces:**

- Molecular level attractive forces
- Energy: in the order of 0.1 eV ( $1 \text{ eV} \approx 1.602 \times 10^{-19} \text{ J}$ )
  - decreases with the sixth power of the distance between molecules
- Larger range compared to chemical bonds
- Hamaker estimated the attractive force,  $F_{VW}$ , between a sphere and a plane surface:

$$F_{VW} = \frac{K_H R}{6y^2}$$

- $K_H$  = Hamaker constant
- $R$  = radius of the sphere
- $y$  = gap between the sphere and the plane

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So, one of the most prominent and most talked about inter particle force you know that it is van der Waals Force, van der; Waals is the name of the scientist after which this force name is there. So, it is the molecular level attractive forces, this van der Waal forces is the forces that are attractive in nature at the molecular level.

The energy level of these forces are in the order of 0.1 eV which is roughly I mean 1 electron volt is roughly equals to  $1.602 \times 10^{-19} \text{ J}$  and this energy decreases with the sixth power of the distance between the molecules. It is the intermolecular forces, it is the attractive in nature, but this strength of this force decreases with the sixth power of the distance between the molecules.

So, as you go far or distant from molecule to molecules this force will strength and drastically decreases, but it is a relatively larger range force compared to the chemical bonds. In a Hamaker estimated this attractive force or identified this force between a sphere and a plane surface. So, how or how much is the intensity of that force? So, if that force is  $F$  this vb van der Waals abbreviated form is vw here we have mentioned, then that force

$$F_{vw} = \frac{K_H R}{6y^2}$$

Where,

$K_H$  = Hamaker constant

$R$  = radius of the sphere

$y$  = gap between the sphere and the plane

So, similarly there are several expressions for this force between the 2 spherical object, 2 planar object, the different expressions are available. So, the gist is that it is attractive force, we have seen it is strength in the order of  $0.1 eV$  this forces, it decreases rapidly as we increase the distance between 2 molecules, but it is still long range force than the chemical bonds. The way of estimation of this force has been established and that has been done by Hamaker well this  $K_H$  is the Hamaker constant in

$$F_{vw} = \frac{K_H R}{6 y^2}$$

So, this is one of the major forces molecular level forces that are there which is relevant for this granulation process.

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**Interparticle forces**

**Forces due to adsorbed liquid layers:**

- layer of adsorbed vapour on the particle surface in the presence of condensable vapour
- overlapping of the adsorbed layers results in bonding forces
- overall strength depends on the area of contact and the tensile strength
- thickness and strength increase with increasing partial pressure of the surrounding vapour
- beyond a critical partial pressure, adsorbed layer bonding superseded by liquid bridge bonding

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The other relevant force is the forces due to adsorbed liquid layers. Now, the layer of say when there is a presence of vapour near the particles then the a layer of adsorbed vapour on the particle surfaces happens in presence of this condensable vapour. On the several we have

say bunch of particles are there and in presence of this condensable vapour we have a layer of adsorbed vapor on the particle surface.

Now, this overlapping of this adsorbed layer results into some kind of bonding forces. Now, overall strength of these forces depends on the area of contact and it is tensile strength. It is thickness thus and the strength of these layers increase with increasing partial pressure of the surrounding vapour. And beyond a critical pressure this adsorbed layer bonding is superseded by the liquid base bridge bonding, these layers then forms the liquid bridges between the particles or the molecules.

So, if we have a bunch of particles surrounded by condensable vapour particle surface adsorbed that vapour and results into a adsorbed layer of that liquid and this results in bonding force because then if these particles are in contact this adsorbed layer basically forms a bridge between these 2. So, the bonding strength of these forces are dictated by the area of contact and it is tensile strength the thickness of this adsorbed layer and it is strength it increases with the increasing partial pressure of the surrounding vapour and beyond a critical pressure this absorbed layer forms the liquid bridges.

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**Interparticle forces**

**Forces due to liquid bridges:**

- presence of miniscule liquid on the particle surface alters the interparticle forces:
  - increasing particle–particle contact due to smoothing effect on surface imperfections
  - reducing the interparticle distance
- presence of sufficient liquid on the particle surface: formation of interparticle liquid bridges
- various liquid states depending on the amount of liquid between groups of particles (Newitt and Conway-Jones, 1958)
  - pendular
  - funicular
  - capillary
  - droplet

Now, as we have introduced now the liquid bridges, this basically happens in presence of any minuscule amount of liquid say on the particle surface, it actually alters the interparticle forces. So, presence of any minuscule amount or this very small amount of water or liquid on

the particle surface alters the interparticle forces, because what it does it actually smoothen the particle roughness effect it is smoothen the particle the surface imperfections.

So, it increases the particle - particle contact and reduces the inter particle distance. But if we have the presence of sufficient amount of liquid on the particle surface, then that previously discussed this earlier discussed forces the magnitude of that does not is not taken into further consideration it becomes insignificant then what happens, it forms the liquid bridges.

Now, various stages or states of this liquid bridges has been identified by researchers and those have been categorized based on the saturation of the inter particle Forces by the liquid phase. We have seen or heard about the term called the porosity or the voidage, now inter particle pore or the space we say the voidage or the porosity. Now, the saturation is the filling up of those forces by the liquid phase or by the liquid. Now, based on the level of saturation we have different state of this liquid bridges starting from pendular, funicular, capillary, and the droplet, this has been identified by the researchers.

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**Interparticle forces**

- pendular:
  - liquid is retained as a point contact between particles
  - separate and independent liquid bridges
  - surface tension forces of the liquid bind the particles
  - capillary pressure resulting from the curved liquid surfaces of the bridge

$$p_c = \gamma \left( \frac{1}{r_1} + \frac{1}{r_2} \right)$$

- pressure in the liquid bridge < ambient pressure: capillary pressure and surface tension boundary attractive force are additive
- funicular:
  - liquid amount increased
  - free movement of liquid
  - reduction in attractive force between particles

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So, how the or what are those stages? And what happens there? How it looks like? So, this is the state we call the pendular stage or the pendular state. Now, what happens here? Liquid is basically retained as a point contact in between the particles in the form of a liquid bridge.

So, these are say the particles this empty circles and in between we have the point contact of the liquid which acts as a liquid bridge. So, liquid is basically stuck in between these 2

particles which in fact act as a binder. So, in case of pendular state these bridges are all separate and independent, because of very low saturation of the or low amount of liquid present in the voidage or interstices.

The presence of this minimum amount of liquid that forms the bridge or forms the point of contact that results in the surface tension forces which binds the particle, this is this liquid surface tension force actually binds the particles and since the bridge is formed there is a curvature of this surface this liquid surface, which results in the capillary pressure. And the capillary pressure is measured as

$$p_c = \gamma \left( \frac{1}{r_1} - \frac{1}{r_2} \right)$$

where  $r_1$  and  $r_2$  are the radius of the curvature of the surfaces and this is the surface tension.

So, when the pressure in this liquid bridge is less than the ambient pressure then this capillary pressure and the surface tension force becomes additive, this becomes this together becomes to the total resultant force. So, this is the initial stage of the say granulation or this formation of the liquid bridge, now why this is important we will see later in granulation. So, here what is the stage that if we have a amount of liquid that is present in the surface the cluster of the particles, the voidage or the interstices the forces are initially filled with the liquid and how it feels or what are the amount of liquid that dictates all these stages.

So, minimum amount of liquid point contact between the particles is the pendular state where we can identify the each and every bridges separately. As the amount of liquid is increased there will be some free water that can move or the free movement of liquid whenever it is present it moves to the funicular structure or funicular state and that reduces in the attractive force between the particles, with further increment in the amount of liquid we move to the capillary state and on enhancing the liquid amount further we get the droplet state. So, basically you can think of this transition of one state to other in forming the liquid bridges is the saturation of the void spaces. The amount of liquid that are present in the force of the particle inter particle force.

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**Interparticle forces**

- Increasing amount of liquid: completely filled interstitial pores results in capillary state
- granule strength decreases due to fewer curved liquid surfaces and fewer boundaries for surface tension forces
- droplet state: particles completely dispersed in the liquid, very low strength
- increasing liquid in pendular state
  - small effect on the strength until the funicular state
  - increases the bond resistance to rupture
- pendular bridges yield in strong granules
  - amount of liquid is not critical
  - should not transit to funicular and capillary regimes
- such saturation can also be increased
  - reducing the voidage/porosity
  - moving the particles closer together

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So, as we increase the amount of liquid it completely fills the interstitial forces resulting in the capillary state. The granule strength decreases due to there are now fewer curved surfaces. So, the capillary pressure and all these are only the important fewer boundaries are there that were responsible for the surface tension forces. So, as you increase the liquid amount it becomes in the capillary state, you further increase the liquid amount it goes to the droplet state. Now, in the droplet state particle is completely dispersed in the liquid. So, quite; obviously, the strength of having of this bond of this force is very low.

So, it has been seen that increase in the amount of liquid in it is pendular state has a small effect of the influence on the strength until it moves to the funicular state. So, if it is not in the funicular state and it is in the pendular state you increase the amount of fluid in the pendular state, it increases the bond resistance to rupture, but as it moves to the funicular state the strength decreases. Now, this pendular bridge yields in strong granules because this state if it is followed in the granulation it results in strong granules, because the amount of liquid is not what the critical condition provided, it should not transit to the funicular or capillary regimes.

The amount of water in the pendular state is not very critical until and unless it reaches the funicular of the capillary regimes, because increasing the amount of liquid in the pendular state until a critical point increases the bond resistance to rupture. So, therefore, it finds relevance in the granulation process because in granulation what we try to achieve is that fine particles or smaller particles and it is agglomeration using agitation as sturdy as possible that

aggregate should be able to withstand certain amount of attrition or external forces in order to not break again. So; that means, as I mentioned you can think of this transition from one regime or one state to the other state as a function of saturation of the liquid phase.

So, such saturation can also be increased by reducing the voidage or the porosity or the moving the particles closer together which is called the densifying operation or concentrating the whole particles together. So, and that is why we have seen these steps in terms of the forces that are acting for the liquid bridges.

So, which means we have now introduced in this class is that the difference of intermolecular forces or the molecular level forces that are relevant for the granulation operation. The reason we have choose only granulation to focus on for the time being is that is it is extensive application is it is popularity.

We will continue this discussion on the different molecular level forces, different surface forces in the next class, because there are couple of forces that are left before I discuss it is relative importance. How these forces are comparable in terms of particle size or when this forces to be considered in which stage, before that we have to go for we have to see the, what are the forces are there; what are the relevant forces are there. So, we will continue this discussion in the next class as well and till then.

Thank you for your attention.