

## Lec 7: Introduction on Size Enlargement

Hello everybody! Welcome to this massive open online course on solid-fluid operations. So in this lecture we will discuss about the size enlargement, its introduction, main categories of size enlargement, applications and as we already discussed about that in the previous two modules what is the particle characteristics, how that particle or solid and fluid can be interacting to yield a certain process yield or process output and also we have discussed that what is the different characteristics of the solid particles and then we have discussed how to reduce the size of the particles to get the more interfacial area and what are those mechanism to get that size reduction. Now in this module also we will discuss something that is about that how to increase the size of the particle that is basically a size enlargement process. So here in this lecture only introduction of that size enlargement will be discussed and the successive lecture we will discuss about its mechanism and also what are the equipments that are being used for this size enlargement here. And here this main question is that what is size enlargement, where that size enlargement process or why we are going to do the enlargement of the size, this is main thing here. So size enlargement is the process by which smaller particles are put together to form larger masses without changing its original particle identity.

So it is called particle or particle size enlargement. It is mainly associated with the pharmaceutical, agricultural, food industries, minerals, metallurgical and ceramic industries. There you will see that sometimes you need to have the granular forms of the powders to deliver it to a particular customer demand. So there in this picture you will see that some powder it will become that granular forms, whereas the powder will have the particle size of micro or nano size.

So after converting into its granular form you will see that size of that particles will be increased. That means here the individual particles come in together and making agglomeration okay or cluster you can say and then giving a certain shape of that agglomeration. So this is basically the process based on which you are getting that size enlargement. But for this size enlargement of course you have to add some additives so that there should be some use of binding of that particles in the powder to get its granular form. So we will be discussing one by one all those things.

So why size enlargement important? You can say that if you are getting that enlarged size of that particles that cannot be said that it will be very huge size. Of course it will be limited or control size of that materials or powders to form its useful one. So to reduce the caking and lump formation sometimes it is required to make that particle into a coarser size. And also creation of non-segregation mixers of ingredients there. Sometimes you will see that some particles will be always stays in segregate mode that cannot be intermix or from that ingredients sometimes it is required to bind together and making a that effective size of that particles for a specific use.

So in that case you have to combine that non-segregating particles with some binders to

form its particular size. So that is why it is required to that size enlargement for this purpose. Now production of useful structural form sometimes you will see that you need to have some structural form of solid materials either to give in a deliberate form or for packing or some other useful manner for which that is required. So in that case you need to have that enlargement of the size and also you will see that sometimes you need to improve the flow properties. If you are having more finer particles you will see that there will be frictional resistance will be higher whereas if you are getting the coarser particles to deliver to a or transport from one location to another location either through conduits or by that build conveyor or some other means there itself it is required sometimes that enlarging that size which will be very that easier to transport that material at that particular size of that material.

In that case frictional resistance may be reduced for that coarser particles and energy consumption will be less. So that is why you need to improve that flow properties just by enlarging the particle. So it is sometimes required for the transportation of the material as well as that some other characteristics suppose flow reactors there also to operate that continuous process by movement of the solid particles there. So in that case for some optimum particle size is required to get that better yield for the process. So there also it is required to enlargement of the particle size.

Then to provide a definite quantity of that active ingredient for a particular you can say that use like that to make the tablet you have to give some definite amount of ingredients for making that tablet. So whenever you are giving that definite amount of ingredients to make that tablet you have to have some optimum size of that particles you cannot use that very finer you cannot use even very coarser particles. So optimum size is required so it is designed as per that ingredient properties as well as other physical properties of the material. And then to improve the product appearance also sometimes you will see that for very fine particles that appearance of the particles will be different from coarser particles. So for coarser particles sometimes you will see that it looks very good or appear nicely other than fine particles there sometimes it is required to enlarge that size.

Also to control that porosity and hardness it is also required to get the optimum size in that case from finer to coarser particle is required. Also to increase the bulk density of the materials for storage it there so you cannot store that particles very finer form rather than that coarser one. So in that case you have to have that optimum size of the particles. Also control of surface to volume ratio in a catalyst supports that is also required for a particular chemical reactions you need to have that optimum size of the catalyst particle there itself it is required to increase that particle size. Sometimes you will see that nanoparticle sometimes it will be coated with some other polymeric substance to give it the better activity in the chemical reaction.

Sometimes nanoparticles cannot be in the solid form used in that chemical reactions maybe in the liquid it will be desolated but to get that crystal or the solid form of that

nanoparticles you need to have that increment of the size just by coating that materials by some other substances. So in that case enlargement of the size is important. Now control of solubility also important here you can control that solubility by changing the size of the particle. Now where this size enlargement can be applied let us discuss one by one here one application it is given that pharmaceutical industries you will see that size enlargement is used to manufacture tablets especially granulation is one of the size enlargement process that is utilized in order to improve both flow and compression characteristics. Now agglomeration is used in pharmaceutical products for them not to contain to have low amounts of dust to provide increased safety during the handling and processing of toxic or medicinally active materials.

So in pharmaceutical industries this size enlargement is important. In chemical industries you will see that also that agglomeration can act as a method to enhance a product providing desired size distributions or product geometry for increased functionality better consumer perception or that protection of the end user from such hazardous or as dust and other possibly toxic or hazardous effects. Sometimes you will see that very fine particles that hazardous effect will be there which cannot be controlled by simple way but there sometimes that very fine particulate materials to make it coarser one you can that separate it from the other particles and then you can separate those toxicity or toxic material or hazardous material from the mixture. So in that way you can protect the users just by converting into higher size material to segregate that hazardous material. As an example is that pigments you will see that since they tend to be dusty and exhibit unfavorable flow and bad metering characteristics there.

So for that you need to have that optimum size of that material. They are often micro agglomerated thus they become dust free smoothly flowing and withstand handling and shipping without degradation. So here you need to have more control of that size just by enhancing its size from its fine particles or nano particles or micro particles just by agglomerating. But you will see that again that size of the agglomeration will be micron size so it is called the micro agglomeration. So that micro agglomeration can be produced from that nano size and it can be controlled where you can get that dust free or smoothly flowing material where you can easily ship without its degradation.

In food industries you will see that application in food processing are you will see that numerous there and are becoming increasingly important more because there you will see sometimes you will see that some structured foods you are actually preferring. So for making that structured food sometimes to prepare it you have to increase the size of the food ingredients and also making a certain shape with that food ingredients. So size enlargement applications you will see that they are in food industries are generally used in the process where different aims such as improving handling and flow ability, reducing dusting or material losses, producing structural that is very useful in forms and also enhancing the appearance etc. Size enlargement you will see that through agglomeration is used to manufacture instant food products thus you will see that it decreases density where

the size of the agglomerates usually ranges from 0.

2 to 2 millimeters. So in that case instant foods reduce drudgery and time preparation and induce improve functional properties like you will see that quick rehydration like that. And also it is widely used in animal feed industry, size enlargement like by agglomeration which is used in the animal feed industry to improve flow, storage, transportation, metering and feeding behavior and also prevent losses and dust annoyance. It is also by agglomeration is used in animal feeding, in veterinary medicine for the you know treatment of sick animals. Also this enlarged size product is a replacement for whole milk in which the butter first removed for mostly human consumption is replaced by a cheaper animal or vegetable fat. So in this direction this enlargement of the size of the particle is important.

You will see that in mining and metallurgical industries sometimes that processing of the iron ore from where that steel is made, so in that case enlargement of iron ore through agglomeration is sometimes required. So iron ore is actually preferred in the form of pellets rather than particles since the configuration of iron ore powder in the blast furnace where that steel is produced is more tightly packed and restricts the air flow. And also you will see that size enlargement through weight agglomeration there are different types of agglomeration you will see that mainly two types, one is wet agglomeration and then dry agglomeration. And then we will discuss the mechanism of that wet and dry agglomeration in the next lecture. So the size enlargement through wet agglomeration and after that it is used to you know combine metal ore you can say that metal bearing powders by dry mixing to yield that formulated alloying components for the final products to become a dust free and easily handleable granular products in the steel industry.

And then application also in the ceramic industries the size enlargement is required, they are like they are doing that size enlargement by agglomeration. In this case you will see that to make this raw materials with the additives for making that ceramic material you need to mix with that additives to make it fillet forms or granular forms for their storage as well as handling properties of ceramic raw materials and additives. Also you will see that these agglomerations will enhance the fine powders, flow properties and packing efficiency. In agricultural industries you will see that size enlargement of fertilizers through granulation is mainly used for getting or ensuring that free ability, freedom from caking and improved that agro-technical properties of the fertilizer. You will see that in fertilizer industries you will see sometimes whatever molten urea is actually produced that actually is converted to granular form in a fluidized bed.

You will see that molten urea is passed from top of the fluidized bed as a molten form and it is sprayed in a cooled air which is supplied from the bottom of that fluidized bed and you will see at a certain temperature control and pressure those sprayed molten urea will be converting into a granular forms in a cool atmosphere of that air supply. There then that whatever form that is called granular form that is actually supplied for the commercial use. So that is why in the fertilizer industries the granulation process is very important

especially at the final stage of that urea making. And then in construction industries of course we are seeing that particulate solids such as sand and naturally occurring binders such as clay are generally mixed and formed or enlarged into a rectangular shapes which is dried and then later fired to obtain really unusable building materials. Then cosmetic industries you will see that size enlargement is used in the cosmetic industry to enhance the appearance of the product.

The particle size to create purely and shimmering looks affect that degree of you will see that glimmer the products, okay? And also the smaller the particle size the less lustrous the powder will be there and the more coverage it gives thus that larger particle size is preferred when made into larger masses in that application. So, once it gives a glitter luster and also is more transparent. So that is why in cosmetic industries that enlargement of the size is important especially for enhancing the appearance of the product. Then you will see that some other things that whenever you are going to enlarge the size from the powder material to make into a granular forms. Some you will see that characteristics of that materials will be considered out of which you can say that some will be it is called interfacial bonding where you will see that whenever granular forms will be there or granular will be made from that binding of individual solid particles or fine particles there it is required some binding agent it is called binders.

We will discuss that binders will show that different types of binders there and by that binders you will see that the particle-particle interaction will be you know enhanced and there are some interaction force will be acting between the particles or among the particles in a powder based on which you will see that this size enlargement will be there or granular form will be made. So what are those interfacial bonds or bonding forces that you have to know? So there you will see that five mechanisms are responsible for making that granulars or size enlargement where inter-particle forces are important that mechanism responsible for inter-particle forces that operating during and after agglomeration. But in this case generally you have to have the stable agglomeration process just by controlling that different bonding forces. What are those forces that you have to know? Like one is called Van der Waals forces and then forces due to it is called adsorbed liquid layers and then liquid bridges and electrostatic forces and solid bridges. These are five types of that mechanism coupling with that forces will be important for that granulation process to know.

So here we will discuss those forces how they are acting on that particles. Then you will see that main forces are Van der Waals forces, forces due to adsorbed liquid layers, forces due to liquid bridges, electrostatic forces, forces due to solid bridges. Now Van der Waals forces that they are exist molecularly based attractive forces between all solids that is called Van der Waals forces and in this case there will be some energy of these forces which will be in the order of 0.1 electron volt and which this is decreases with the sixth power of the distance between molecules and the ranges of Van der Waals forces is large compared with that of chemical bonds. So they are the particle-particle bonding mainly because of

these Van der Waals forces and based on which that agglomeration will get its stabilized form to give you that particular shape of that granules.

So here that attractive force between solids will be mainly dominating by that Van der Waals forces. Now that attractive force generally represented by this FVW it acts between a sphere and a plane surface as a result of Van der Waals forces which is actually derived by Hamaker and is usually presented in the form like this. So FVW will be equal to  $K_H R_1 R_2$  divided by  $R_1 + R_2$  into  $6r^2$ .

Here the equation(s)

$$F_{VW} = \frac{K_H R_1 R_2}{(R_1 + R_2) 6r^2}$$

$$K_H = (0.4 - 4.0) \times 10^{-19} J$$

Here  $K_H$  is called the Hamaker constant. It has a strong correlation with the various physical phenomena such as liquid wettability, adhesion, friction, adsorption, colloidal stability, polymer flow and also deformation of the particles.

And its value generally ranges from 0.4 to 4.0 into  $10^{-19}$  joule. And other terms like  $R_1$  and  $R_2$  these are the radius of the particles or spheres in the powders and then small  $r$  square here it is basically the gap between two particles or the spheres. So in this way you can easily calculate what will be the Van der Waals force there in the agglomeration or agglomeration or granules whenever it will be formed there.

Now forces due to adsorbed liquid layer is another important points there for making that granules or for enlargement of the size. particles in the presence of a condensable vapour will have a layer of adsorbed vapour on their surface. Here one particle if this particle is placed in the condensable vapour you will see that there will be a layer forming surrounding this particle. And you will see that the strength of the bond will be depending on the area of contact and the tensile strength of the adsorbed layer. So in this case one layer will be formed adsorbed liquid layer it is called on the surface and the strength of that bond will be depending on the area of contact and the tensile strength of the adsorbed layer.

If these particles are in contact bonding forces which will results from the overlapping of the adsorbed layers. And the thickness and strength of the layer increase with increasing partial pressure of the vapour in the surrounding atmosphere on which you are placing that particle or powder. According to Kuelho and Hernby you will see that in 1978 they are actually stated that there will be some critical partial pressure at which the adsorbed layer bonding gives way to liquid-breeze bonding. So that means there are two particles if come in contact so between that particles there will be a adsorbed liquid layer and over which there will be some partial pressure. So there will be some critical partial pressure at which this adsorbed layer bonding between these two particles gives way to liquid-breeze

bonding.

That means there will be a liquid-breeze between that particles. So that bonding will be happened only beyond this critical partial pressure. Then forces due to liquid-breezes. Now whenever that liquid-breezes will be formed like this here two particles and between that there will be a liquid-breeze. Now you will see that in addition to that inter-particle forces that is Van der Waal forces there another forces that will result from this adsorbed liquid layer.

The presence of liquid on the surface of the particles that affects the inter-particle forces by the smoothing effect on the surface or by increasing particle-particle contact or by its effect of reducing the inter-particle distance. Now these forces are usually negligible in magnitude compared with forces resulting when the proportion of liquid present will be sufficient to form that inter-particle liquid-breezes. So here what are the forces that maybe that some surface tension forces, capillary pressure forces there. So these forces you will see that maybe that ineffective in magnitude compared with the this bonding forces of making liquid-breezes. Now forces due to liquid-breezes in the case of different form of that agglomeration, you will see that different types of orientation of that binding liquids which will give you that different shape or different states, okay different states of that formation of that or binding states of that particulate materials by that binding liquids or binding agents.

And in this case Newitt and Conway Jones, 1958, they have identified 4 types of liquid states depending on the proportion of liquid present between that means group of particles. And these states are known as like this one is called pendular states, another is called funicular states, another is called capillary state and then droplet state. So these are different types of liquid states will be there based on the proportion of liquid present between groups of particles, okay as shown in the picture here. As the proportion of that liquid to particles is increased, you will see that the liquid will be free to move and the attractive force between particles will decrease. When there is sufficient liquid to completely fill the industrial pores between the particles, specially that capillary pores, the granule strength will fall down and it will fall further as there are fewer curved liquid surfaces and fewer boundaries for surface tension forces to act on.

And you will see that when the particles are completely dispersed in the liquid that will come as a droplet form, the strength of the structure will be very low. And in case of pendular bridges, here this pendular bridge you will see that this pendular will give you the strong granules in which the quantity of liquid is not critical but should be less than that required to move into the funicular and capillary states. Then another important force which is acting on that liquid and liquid which is forming, liquid and particles or particle and particles, you will see that this electrostatic force, you will see that this electrostatic surging of particles and surfaces sometimes occurs as a result of friction that is caused by inter-particle collisions and frequent rubbing of particles against equipment surfaces

during processing. So whenever particles will be used to make it granular forms, there will be some interaction between particles, there will be some attrition between particles, there will be some collision between particles. So during that particles collision, there will be some that electrostatic surge will form on the surface.

As a result of that friction or collision caused by that inter-particle collisions and frequent rubbing of that particles against that equipment surfaces during the process, the surge will be caused by the transfer of electrons between the bodies. The force between two surge spheres will be proportional to the product of their surges. Now this electrostatic force says may be attractive or repulsive, that do not require contact between particles and can act over relatively long distance that compared with it is called additional forces which require the contact between the particles. So here one important point that the electrostatic forces also will be one important component to bind the liquid or make the granules there. And it is happened because of that collision between the particles, rubbing of that particles with the surface of the equipment.

And also during that collision, there will be a transfer of electrons between the bodies and because of which you will see that there will be electrostatic charging. And this charging forces or electrostatic forces between two charge spheres will be proportional to the product of their charges, which can be expressed by this equation here. What will the charge forces will be there? That will be is equal to  $K \frac{Q_1 Q_2}{R^2}$ . Whereas  $Q_1$  and  $Q_2$  are the charge density and  $R$  is equal to distance between two charged particles. And then another important point during that granulation process or making the enlargement of the size, in that case it is called solid bridges.

Sometimes solid materials will make a bonding between two particles. In that case granules formed by liquid bridges are usually not the end product in this case. We will see, whereas you will see that more permanent bonding within the granule will be created or will be enhanced by solid bridges that is formed as liquid which is removed from the original granule. I think you understand here, see in the picture there is a solid bridge.

Here there is no liquid bridges forming. Okay? So finally this solid bonding will be there, solid bridge will be making to make this agglomerates. Solid bridges between particles may take three forms. One is crystalline bridges, another will be liquid binder bridges and then solid binder bridges. So this is the solid binder bridges and if the material of the particles is soluble in the liquid added to create granules, the crystalline bridges may be formed when the liquid evaporates. Okay? So it is important that crystalline bridges will form when the liquid will evaporate there.

Some binding solvent will be used as which is very that volatile. So when after bonding of that liquid bridge and then finally solid bridge you will see that some binding agent that is which is very volatile it will be evaporates and then after evaporation that crystals forms of that granules will be there. And the relative magnitude of the different bonds as a



function of particle size can be interpreted or can be represented by making a map like here shown. In this case you will see that if we consider the tensile strength of suppose granules after making bonding or size enlargement or making granules, what will be the size of that granules? Let it be considered as a particle diameter. So in this case if you represent this tensile strength versus particle diameter, you will see at a certain range of particle diameter the tensile strength will be prominent or to get the less effective tensile strength you have to produce a certain range of particles to have the optimum size of the granules just by controlling the different forces whenever they are making a bond.

So in this case, in this map it is shown that the tensile strength versus particle diameter and there are some region it is shown here, this region is it is called Van der Waals that means in this region within a size range of this particle and this range of this you know tensile strength, here the Van der Waals force will be dominating compared to the other. Whereas within this region you will see that Van der Waals in the presence of adsorbed liquid layer should be dominating there and in this region here only liquid bridges will be effective whereas other forces will not be effective there. And then beyond these three regions there will be another region it is called solid bridges. So that solid bridges will be formed only within this particle range, particle size range and the strength of the at the strength of the material. So here we can say from the combination of this tensile strength and the particle diameter which force will be dominating and which force will not be dominating or effective that can be assessed.

So Van der Waals force will become important only for particles below 1 micron in size that you have to remember and adsorbed vapour forces that will be very relevant below the particle size will be 80 microns. Whereas the liquid bridges forces are would be more effective or active you can say below about 500 micron size particle. And then you will see that we are talking about here the size enlargement there should be a certain process to make this size enlargement. There are different process available though all the process are will be of same mechanism only thing is that some terms will be used to represent the operation of that size enlargement by different types of industries. So there are different terms of that size enlargement process like called compaction, it is called granulation, sometimes it is called encapsulation, sometimes it is called pelletizing and sometimes it is called agglomeration.

So these actually terms are basically the same that size enlargement but as per industry brand they are giving the different terms but they are basic mechanism will be same for that size enlargement. So here compaction basically you will see that the process involving large deformations, large strain, non-linear material, behaviour and friction where granulation whenever you are talking about it transforms a powder material into larger entities to end up with an agglomerate or you can say aggregate considerably larger in size and with a porous structure. Whereas encapsulation it is in you will see that pharmaceutical industry or pelletizing in the pharmaceutical industries, in the encapsulation it is said that this process whereby various ingredients can be stored within

a specific size shell or coating for protection and later release. And pelletizing in this case it is the process of compressing or molding a material into shape of a pellet. And then agglomeration it is basically the formation of assemblage in a suspension and particles dispersed in the liquid phases stick to each other and form irregular particle clusters, flocks or agglomerates.

So these are called agglomeration. So there are several processes almost the same mechanism but their name is different. So in this case we will discuss only that as per that syllabus, the undergirded syllabus we will discuss some mechanism of granulation process in the next lecture. So before completing that we are coming to the some point here that some you will see that some binding agents to be used for that agglomeration process to make that granular forms. So in this case it is called excipients. Excipients for granulation can be largely divided into categories like bulking agents, functional additives and others like coolants, coating aids, stabilizers, pH modifiers and release rate modifiers.

And bulking agents or sometimes it is called fillers, okay? So fillers serve to form the core or structure of a dose's form. And functional additives include binders, disintegrants, that lubricants, colorants and stabilizing agents. And these bulking agents generally differ from the functional additives in that they are usually inert materials that are relatively very inexpensive. The choice of excipients depends on the number of factors like drug, which drug you are going to use, the process that you are following and the formulator and also the matter of cost. So bulking agent generally common bulking agents are sugar, lactose, starch, microcrystalline cellulose, dicalcium phosphate like that.

Some functional additives like binders, disintegrants, lubricants, stabilizing agents, colorants like that. Now binders may be wide varieties, some maybe will be sugars and some will be maybe polymer and some natural polymer, some will be synthetic polymer, even semi-synthetic polymers are there. Sugar like sucrose, glucose, sorbitol, those are used for binders. Natural polymers like acacia, alginate, sodium alginate, gelatin, etc.

are used. The semi-synthetic binders like ethyl cellulose, sodium carboxymethyl cellulose, these are being used. Synthetic binders like polyvinyl, pyrrolidone, they are used. Polyethylene glycol generally used. Natural binders like xanthan gum, you will see that xanthan gum, those are being used. Others like isopropyl alcohol, chloroform, those are also used as a binder solution.

And why that binder solutions to be used there? The main role of binders is to provide the cohesiveness which is very required for the binding of the solid particles under compaction to get the certain shape of that granules or tablets or some other forms. You will see that binders also improve the hardness of the tablets by enhancing intragranular as well as intergranular forces. Also you will see that the cohesive properties of the binders reduce the free ability of the tablets and also like polyethylene glycol which is used as a binder is in a melt granulation which is very important in both wet granulation and fluid bed granulation.

So they are, those are actually very important to give a certain cohesiveness force to give a controllable size. And as per that scope of that level of course, the details of granular process will be discussed in the next successive lectures and what is the mechanism and what are the basic equipments will be used for the granular process will be discussed in the next lectures. So thank you, have a nice day.