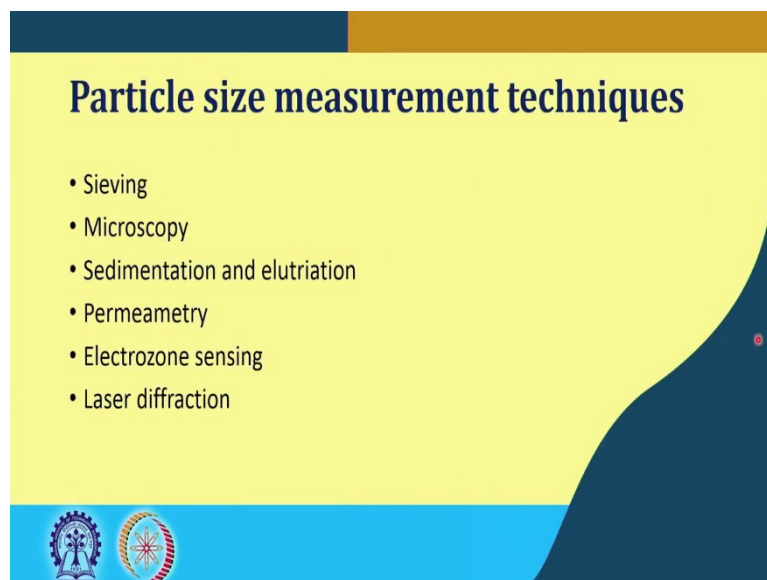


Fundamentals Of Particle And Fluid Solid Processing
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Lecture - 02
Solid particle characterization (Contd.)

Hello everyone. Welcome back to the another class of Fundamentals Of Particle And Fluid Solid processing. Today we will go through the Solid particle characterization that we started on day 1.

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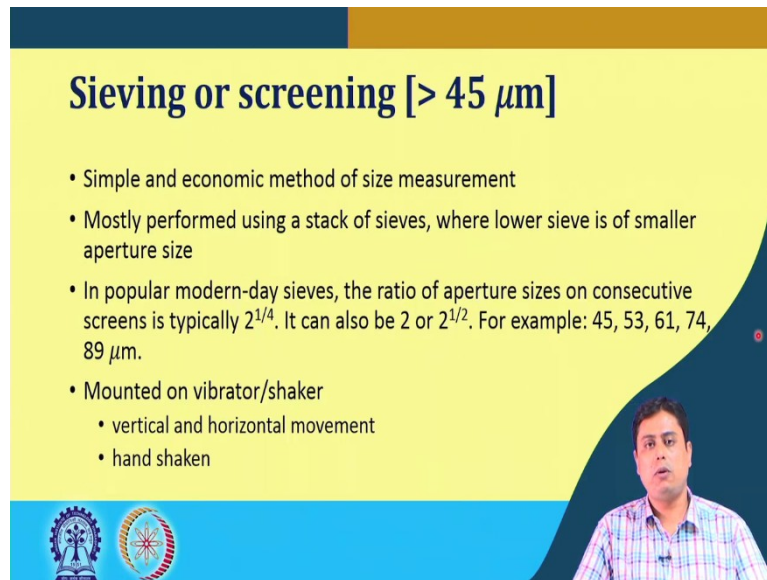
At the end of day 1 I mentioned that particle size measurement techniques will be covered in this class, those I mentioned the names as well are the sieving, microscopy, sedimentation and elutriation, permeametry, electrozone sensing and laser diffraction.

So, in this lecture we will cover this methodologies to see that how a single particle size can be determined or a cluster of particles or a certain amount of particles and its size distribution can be evaluated. So, the point here I want to mention here that although we started with the single particle, but you can understand that in industrial application a single particle size or its a shape is a kind of irrelevant matter because there we handle bulk particulate solids.

So, eventually if that is not a mono sized particle or uniform size particles which is very very difficult to achieve or has rather limited applications, we typically have a wide range of

particle size and shapes. So, that is why particle size distribution becomes important in such cases. We will come to that in due course, but let us at first focus on this techniques the overview of this techniques which will help us to understand that how such particle size and shape sometimes can be determined by these techniques.

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Sieving or screening [$> 45 \mu\text{m}$]

- Simple and economic method of size measurement
- Mostly performed using a stack of sieves, where lower sieve is of smaller aperture size
- In popular modern-day sieves, the ratio of aperture sizes on consecutive screens is typically $2^{1/4}$. It can also be 2 or $2^{1/2}$. For example: 45, 53, 61, 74, 89 μm .
- Mounted on vibrator/shaker
 - vertical and horizontal movement
 - hand shaken

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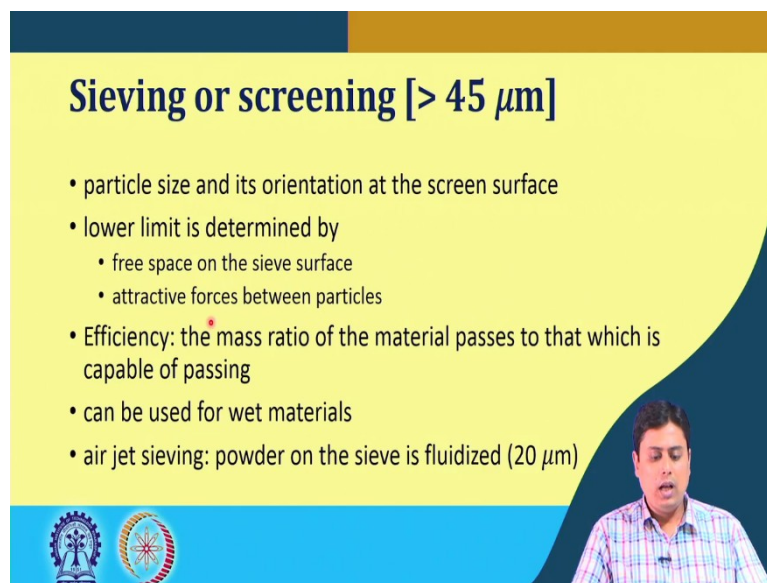
So, first of all we will start with the sieving methodology. In sieving or also it is popularly called as a screening mechanism, it is one of the most simple in fact, the simplest and economic method for the size measurement. It is mostly performed using a stack of sieves where lower sieve is of smaller aperture size. Now, one of the typical example of sieving we see in our household applications during this separations of different size of lets say rice grains or wheat or flower when we do that in a strainer or even when we separate the tea leaves from tea with a strainer.

So, similar kind of principal is applied in this sieving or the screening technology where bulk solids are put into the strainer into basically these are the cascade of strainers or the screens and most cases it is vibrated in vertical as well as in horizontal directions. So, these are the stack of sieves where from one to another from top to bottom where it is firstly, fade there the aperture sizes are bigger and then smaller and consecutively it goes smaller and smaller. In modern day applications and in the popular sieves the ratio of the size apertures on consecutive screens is typically the 4th root of 2, but it also can be 2 or a square root of 2.

So, the popular sizes are as I have mentioned here are these 45, 53, 61, 74, 89, if you see that this ratio 53 by 45 is numerically or approximately close to the numerical value of 4th root of 2. So, there are several standards are available like the US standard, British standard, Indian standard. So, in these cases as I have mentioned here the typical aperture ratio is a 4th root of 2, but when it was initially implemented there was also examples of the ratio could be 2 or square root of 2.

Now, since this is a stack of sieves where the bulk solids are put into the screen, it is typically mounted on a vibrator or shaker where there is option of vertical and horizontal movement or sometimes it is also hand shaken. So, the point here is that majority or let us say some amount of solid particles goes through that aperture, if those are small smaller in size than the aperture size and those are bigger they are retained as overflow of the strainer or the screen. And whichever passes through the screens we typically call the underflow.

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Sieving or screening [$> 45 \mu\text{m}$]

- particle size and its orientation at the screen surface
- lower limit is determined by
 - free space on the sieve surface
 - attractive forces between particles
- Efficiency: the mass ratio of the material passes to that which is capable of passing
- can be used for wet materials
- air jet sieving: powder on the sieve is fluidized ($20 \mu\text{m}$)

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Now, quite naturally you can understand this particle size and its orientation on the surface screen becomes a dominant factor to have a efficient screening mechanism because not every particles are spherical, it is of irregular shape in practice. So, let say the example of this pen. So, if it falls like this on a screen which has a linear dimension of aperture smaller than this one, it can it will basically be retained on the screen, but it falls in this direction in vertical directions it can easily pass through such aperture.

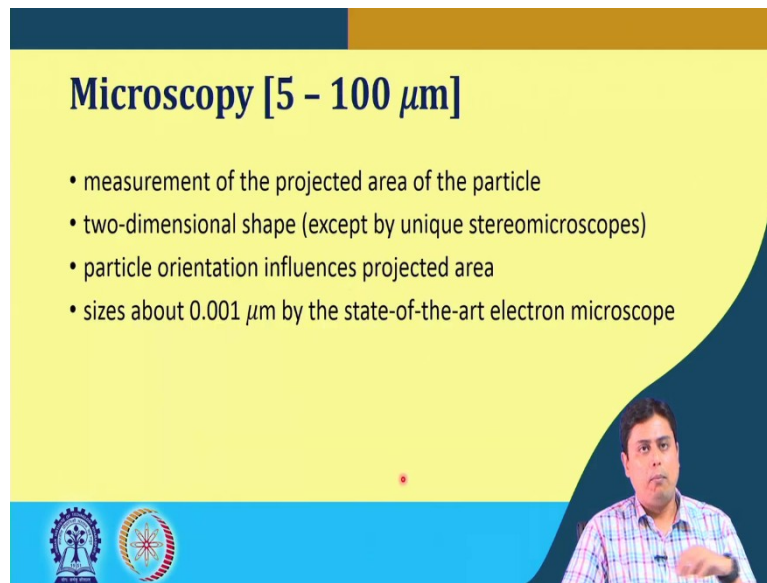
So, the orientation how it falls on the screen actually determine the efficiency of the screening and definitely the particle size. So, whichever passes through we say that this we can understand that the dimension of those particles will definitely be lower than the aperture size. The lower limit of size separation by this process eventually dictated by two mechanisms; one is the free space available on the sieve surface because if you go smaller and smaller in this aperture size there will be a less lesser amount of area available for the particles to go through, which becomes a difficult for the screening process.

And the other thing is that as you go smaller and smaller in the aperture size, the attractive force between the particles becomes dominant the Van der Waal force is becomes dominance dominant. So, it tries to agglomerate and clog the aperture and that is why this vibration or shaking operation helps some times to get very fine particle size that should go through the aperture or its size determination by the size separation. As I said the efficiency of such process which is typically defined by the mass ratio of a material that passes through the sieve to which it is capable of passing because practically the efficiency cannot be is not basically a 100 percent.

So, it may be capable of passing through let's say m 1 kg of a certain size particles, but it is separating m 2 kg of certain size of that size of particles. So, the efficiency is always lesser than the 100 percent. typically such this screening or sieving are used in a dry condition; that means, the dry solid particles are a dumped on the first screen which is then shaken or the vibrated. Its under flow goes to the another sieve which has a lower aperture size and then subsequently it goes through a series of screens. But it also can be used for the wet materials; that means, the suspension the dilute suspensions dense suspension sometimes.

Now, in air jet sieving which is one of the wet material screening where the powder on the sieve is basically fluidized. So, the powder becomes suspended and it flows through the smaller aperture and by this kind of a wet technique, even though and the heading I mentioned that the sieving or the screening operations are typically applied for size of more than 45 micron by weight screening or this air jet sieving, the particle size also can be separated till 20 micron or we can. Once I say that this is a particle size separated for 20 micron; that means, the 20 size micron particles we can identify that the size range.

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Microscopy [5 - 100 μm]

- measurement of the projected area of the particle
- two-dimensional shape (except by unique stereomicroscopes)
- particle orientation influences projected area
- sizes about 0.001 μm by the state-of-the-art electron microscope

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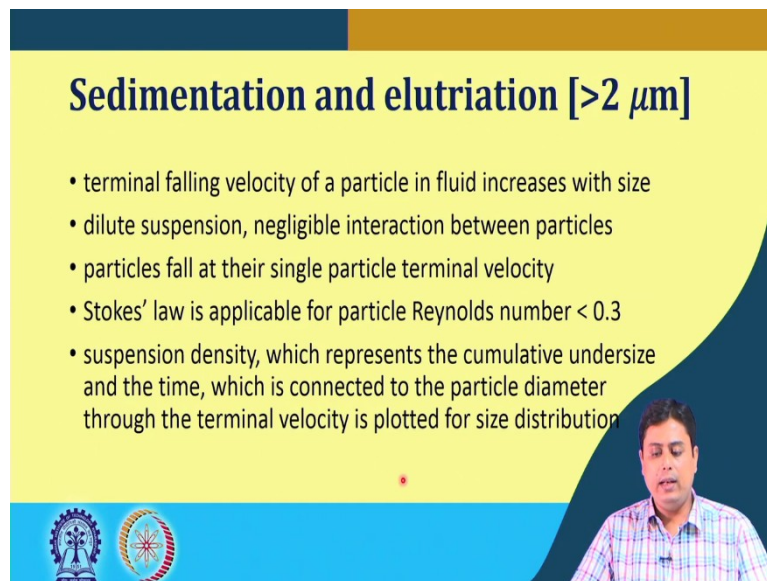
Next if we go for microscopy method in microscopy method that is that you place a particle under an optical microscope. So, here that you can do for single particle you can put it under optical microscope and determine its size or you can put several particles together under the microscope to have a size distribution.

Now, one of the biggest disadvantages of this microscopic method although it is a direct measurement process, the issue is that it gives us the two-dimensional representation of a three-dimensional body because the thing that you are seeing is a projected area of a particle. And if the particle is not spherical or let's say the irregularly shaped particle, then what you see is basically the projected area that you have to equate or correlate with the kind of diameter that I mentioned in my previous lecture, like Martin's diameter or Feret's diameter to have a mean diameter of a single particle or a particular representative diameter of a single particle.

So, the particle orientation as I said during the sieving also that if you see this type of a particle under the microscope you can determine its longitudinal distance, but if you put it like this you can see only the top part of this which is which you would see in a circular or a circular area. So, particle orientation basically influences this measurement process like the sieving also, sometimes you can do it in a random orientation to get an average diameter of a single irregular particle.

By this method sizes about 0.001 micron can be achieved although in the heading I have mentioned that this technique is typically applied for 5 to 100 micron of particle by simple optical microscope, but if you have a state of a electron microscope there you can achieve to very very finer particle size which is almost in the range of 0.001 micron or let's say the 1 nanometer size particles.

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Sedimentation and elutriation [$>2 \mu\text{m}$]

- terminal falling velocity of a particle in fluid increases with size
- dilute suspension, negligible interaction between particles
- particles fall at their single particle terminal velocity
- Stokes' law is applicable for particle Reynolds number < 0.3
- suspension density, which represents the cumulative undersize and the time, which is connected to the particle diameter through the terminal velocity is plotted for size distribution

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The next technique I would discuss is the sedimentation and elutriation which helps us to have the particle size measurement for greater than typically 2 micron. So, these technique are typically applied for particle sizes greater than 2 micron in size. So, here the principle of the sedimentation process is determined or it based on the fact that the terminal falling velocity of a particle in fluid increases with size.

If you remember Stokes' law this is as I mentioned as a prerequisite fluid mechanics knowledge that you need to have for this course you would understand that this terminal falling velocity is essentially dependent on the particle size. In this technique based on that fact we typically apply Stokes' law which is applicable for particle Reynolds number of less than 0.3. And also if you may remember that Stokes' law is applicable for the low Reynolds number cases and it is applicable for dilute suspension where there is negligible interaction between the particles.

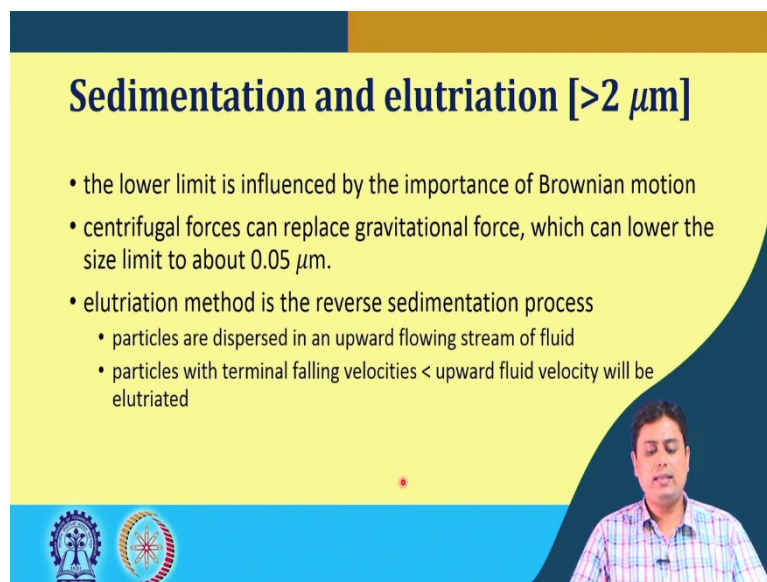
So, that means, we assume that this particles when it is thrown into a pool of liquid or a fluid, it is settling through its single terminal velocity. This particle achieve their single particle

terminal velocity, it happens only when this is the dilute suspension is in consideration. So, quite naturally the suspension density that depends on the cumulative undersize and the time which is connected with the particle diameter through the terminal velocity is plotted for the size distribution and we get the mean size from this size distribution.

So, what happens? Let's say if you take an example you have a suspended liquid dilute suspension, the particles are falling at a certain plane from on the vertical axis you measure the concentration of the particle. And then this suspension concentration or the suspension density is basically what does is the undersize particles, but which are falling with the terminal velocity. And the time it takes to reach that stage is directly related with the terminal velocity. So, we plot this two information, we get its size distribution that the bigger size let say the bigger size particles are falling at a higher rate, the lower size particles will fall at a smaller rate.

By this technique typically two micron particles can be separated, but if we apply Stokes' law in water we will see the calculation shows that typically particle sizes of 50 microns and larger are typically easily determined by the this when the fluid is water.

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Sedimentation and elutriation [$>2 \mu\text{m}$]

- the lower limit is influenced by the importance of Brownian motion
- centrifugal forces can replace gravitational force, which can lower the size limit to about $0.05 \mu\text{m}$.
- elutriation method is the reverse sedimentation process
 - particles are dispersed in an upward flowing stream of fluid
 - particles with terminal falling velocities $<$ upward fluid velocity will be elutriated

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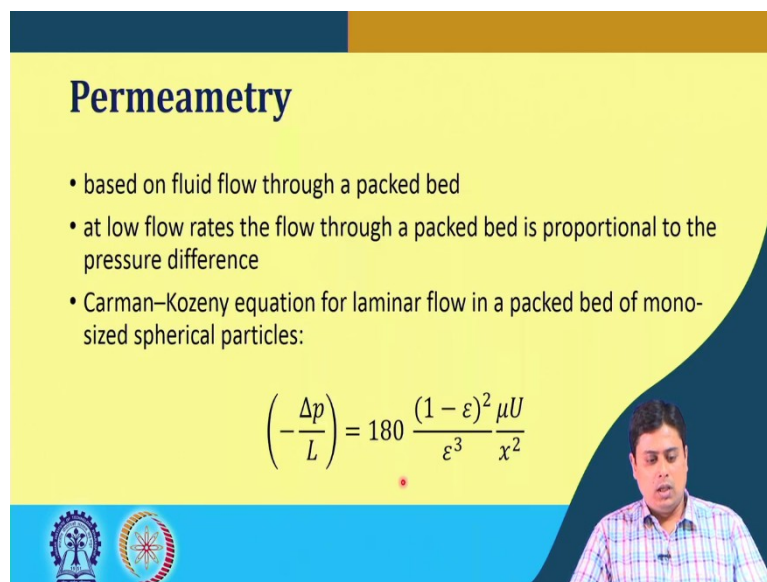
The lower limit in this case is influenced by the importance of Brownian motion because as we decrease the particle size the Brownian motions becomes dominant. So, the lower limit we have to set accordingly or we can determine based on the fluid property that we chose when this particles we are sedimenting.

Centrifugal force can replace this gravitational force like instead of a simple normal settling cases, we can introduce centrifugal forces in that domain to enhance that settling process and in that case the size limit of about 0.05 micron particles can be identified or can be settled in this case. Because we can understand such lower size particles under normal settling conditions may not settle and it can be suspended forever.

But with the introduction of centrifugal force instead of the normal gravitational force we can have very lower size particle determining in this case. Elutriation is basically the reverse of this sedimentation process, where particles are dispersed in an upward flowing stream of fluid. So, instead of now in sedimentation what was happening? The particles were coming from top to down, but in elutriation it is coming from bottom to top and the particles which have a terminal falling velocity lesser than the upward fluid velocity it will be carried away by that flowing stream or will be elutriated.

So, again at a certain vertical plane we measure the suspension density we capture the time and we plot that and we get a size distributions at different time steps that how much is the particles are being elutriated or sedimented in these cases.

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Permeametry

- based on fluid flow through a packed bed
- at low flow rates the flow through a packed bed is proportional to the pressure difference
- Carman–Kozeny equation for laminar flow in a packed bed of mono-sized spherical particles:

$$\left(-\frac{\Delta p}{L}\right) = 180 \frac{(1-\varepsilon)^2 \mu U}{\varepsilon^3 \chi^2}$$

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The next method is the permeametry method. In permeametry it comes from the principle of the fluid flow happening through a packed bed of spheres. Typically this spherical size particles are used in packed bed to simplify this calculation in permeametry.

But if that is of random size and shape that also can be determined we can determine its mean diameter or mean size by this technique. So, what happens here that we simplify the Ergun equation by assuming that the at the flow is happening at a very low flow rate. So, at low flow rate the flow through packed bed is basically a proportional to the pressure difference gradient or the pressure gradient across the length.

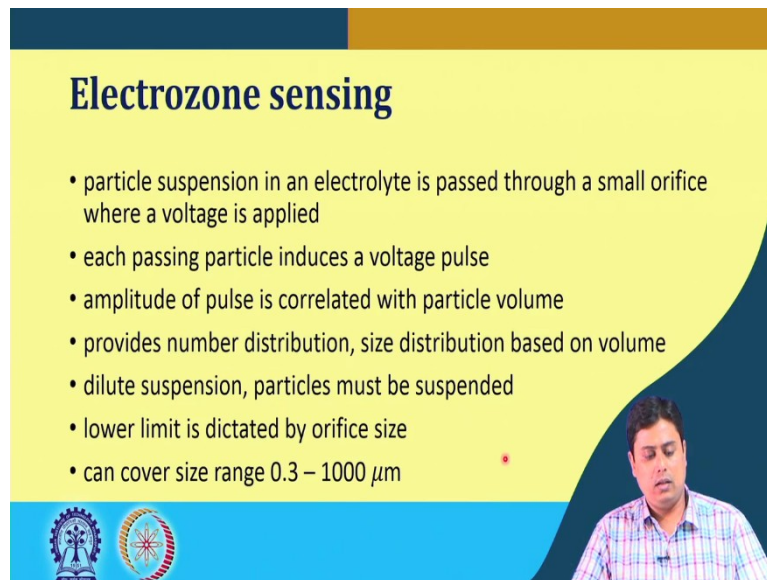
So, typical Kozeny Carman equation for laminar flow in packed bed of mono sized spherical particles is something that I have written here.

$$\left(\frac{-\Delta p}{L}\right) = 180 \frac{(1-\varepsilon)^2}{\varepsilon^3} \frac{\mu U}{x^2}$$

So, here you can see that the $\frac{\Delta p}{L}$, this is the pressure gradient per unit length is proportion or basically has a relation with the particle size to the power 2 or square of the particle size which is inversely proportional to this pressure gradient. So, for a particular flow rate or the U where which is the superficial velocity here which comes from the volumetric flow rate or mass flow rate and epsilon is the voidage or the porosity of the packed bed.

So, for a known porosity or a known porosity of a packed bed of a known fluid property mu you can calculate or you can estimate by experiment that what is the pressure gradient happening through a unknown sized particle. You do it for multiple times, you plot the graph and you can find out what is the value or the mean value of x. Or in fact, if this is the mono sized particle the calculation becomes much more easier that for a particular flow rate you estimate its pressure gradient across the bed, you use this relation and get the value of x which is the particle size.

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Electrozone sensing

- particle suspension in an electrolyte is passed through a small orifice where a voltage is applied
- each passing particle induces a voltage pulse
- amplitude of pulse is correlated with particle volume
- provides number distribution, size distribution based on volume
- dilute suspension, particles must be suspended
- lower limit is dictated by orifice size
- can cover size range 0.3 – 1000 μm

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The next method we will discuss is the electro zone sensing. Now, in this case what happens that the particle suspension in an electrolyte is passed or let's say is withdrawn through a small orifice where the voltage is applied across the electrolyte and that withdrawing chamber. So, what happens that the when there is electrolyte and a constant supply of voltage is there is then the electrolyte impart some resistance.

Now, when a particle passes through that orifice during this withdrawal of the suspension, what happens that the particle actually displaces some electrolyte to go through that orifice and since electrical voltage is applied across that orifice it creates a voltage pulse. So, each passing particles induces a voltage pulse and the amplitude of this pulse is correlated with the particle volume or which is directly is the displacement electrolyte volume which is equals to the particle volume.

By doing this what happens when multiple particles are passing at different time you get a several pulses voltage pulses that you collect and then you process that signal by some algorithm and you get its size distribution and the number distribution; number distribution you can explicitly get as one the by counting the number of pulses. And the size distribution based on volume you can achieve also by this method.

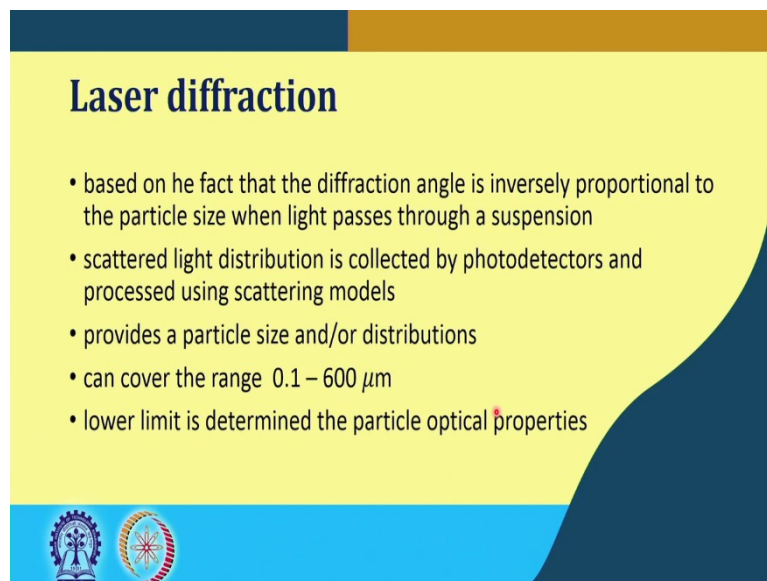
Now, you can understand that it can create problem if multiple particle passes through the orifice at a certain time, then you do not get the actual measure. So, this process has limitation that you have to take a dilute suspension. So, that one by one particle can be

withdrawn to that orifice or can pass through that orifice. The other criteria is that particles have to be suspended, if the particle settles then it is very difficult that to pass it through that particular orifice. So, particle has to be suspended.

Now, this lower limit of the linear dimension that you can achieve by this methodology is dictated by the orifice size that how small your orifice size basically that will be your minimum or the maximum size of the lowest size particle that you can determine. Typically it can cover the size range from 0.3 to 1000 micron size particle you can determine, the higher or the upper limit is dictated by the criteria of as I mentioned that the particle must be suspended, if this is the bigger particle it tries to settle down.

So, you may have to change in fact, you have to change the suspending fluid media you have to take more viscous fluid, so that it can be suspended for a longer time until and unless you withdraw it to the orifice.

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Laser diffraction

- based on the fact that the diffraction angle is inversely proportional to the particle size when light passes through a suspension
- scattered light distribution is collected by photodetectors and processed using scattering models
- provides a particle size and/or distributions
- can cover the range 0.1 – 600 μm
- lower limit is determined the particle optical properties

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The last methodology is the laser diffraction. Laser diffraction what happens? We calculate the sizes based on the fact that the diffraction angle is inversely proportional to the particle size when the light passes through a suspension. So, if you have some fluid particles suspended in a fluid media you through a laser light the light will be scattered when it is incident on a particle; on a particular particle.

So, similarly all these scattered lights is then collected and you process in a photodetector and this scatters were there are several scattering models are available to process those signals, then it provides the size of particle size and its distribution.

So, if there is a single particle in the suspension and laser light is reflecting from there you can determine the single particle or if there is multiple particles suspended in a suspension the collective response will give you the particle size distributions. By this technique one can measure from 0.1 to 600 micron particles the lower limit of by this process beyond 0.1 micron is basically I mean determined by the particle optical property.

If the particle optical property changes beyond typically it changes beyond that limit, but if that does not change for your particle you can use it for the you can even go beyond that particle size, but in some cases even 0.1 micron particle size and let's say the 1 micron particle size will have different optical properties. So, in that case you cannot go beyond that limit after which the optical property of that particle changes.

So, this are the overview of the measuring techniques of a particular for single particle or a chunk of or a mix of particles, we will when will go through in details during the fluid particle mechanics as well as when the fluid is suspending in a pool of liquid we will go into the details or will see the examples how those happened we will be link with this techniques during that study, but for now I will stop here and we will see you with the next lecture.

Thank you for your attention.