Fundamentals of Materials Processing (Part–1)
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Lecture Number 31
Powder Characterization using Gas Permeability Method

So we will continue our discussion on gas permeability for characterizing powder. So you saw we used Darcy's relation from where we get the alpha and using that alpha and the this parameter porosity we can get surface area. So we will see how or the way that porosity is defined, so this is very interesting we use what is called as packman analogy.

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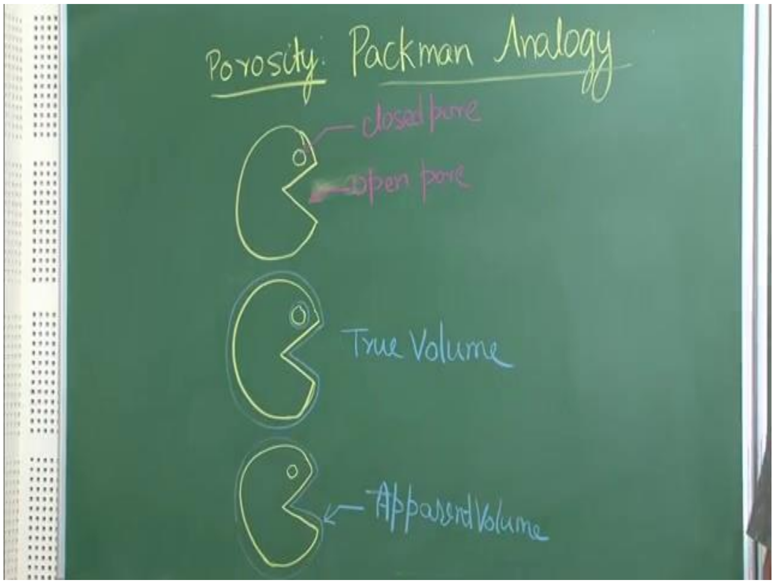

So we are talking about porosity and we will talk about the method which is called packman analogy and if you have ever played the game of Pac Man you would like it, the many of you from the new generation may be more aware of Pokemon Go the game then Pac Man and P ac Man, anyhow let us get back to let say this is a powder particle and it has a pore like this, so this will be called open pore what we are looking at is one particle and this will be called a closed pore. Now what is the closed pore? And how is it different from open pore? Open pore as you can see is through and through the particle, so if the gas is flowing, it will allow the gas to flow all the way through. On the contrary, the closed pore although it comes to the surface but it does not pass through the whole particle, so the gas cannot pass through it.

However, it is still leading to increase in the surface area, because the surface area you can think of some hemispherical scoop cut out from the particle, so there is a surface area increased because there is a pore over there. So that is the difference between a closed pore and the open pore. Now if you were to define different true volume, you would point imagine that the true volume would look like something like this, so there is a pore over here, there is a pore over here, this is a open pore, this is a closed pore and if you want to describe a true volume we will take away this much volume, we will take away this volume, so we are considering only this volume.

So volume inside this and taking away the volume taken out because of that pore. So that will be called true volume, however in most cases it will be difficult to estimate this volume, and therefore what you will be estimating is what will be called a apparent volume. So what you will actually be calculating is you will include this volume only exclude this open pore volume and therefore you will be taking into account only (th) point this whole volume including this volume.

So some amount of volume has been taken, extra volume has been into account and therefore density that you will calculate will be lower than what should be for the true density, so this will be your not true apparent volume. So with these definitions we are now in a position to describe the term porosity that we started using last time. And in fact we should be able to get what is called is true porosity and you will see why we call this as true porosity.

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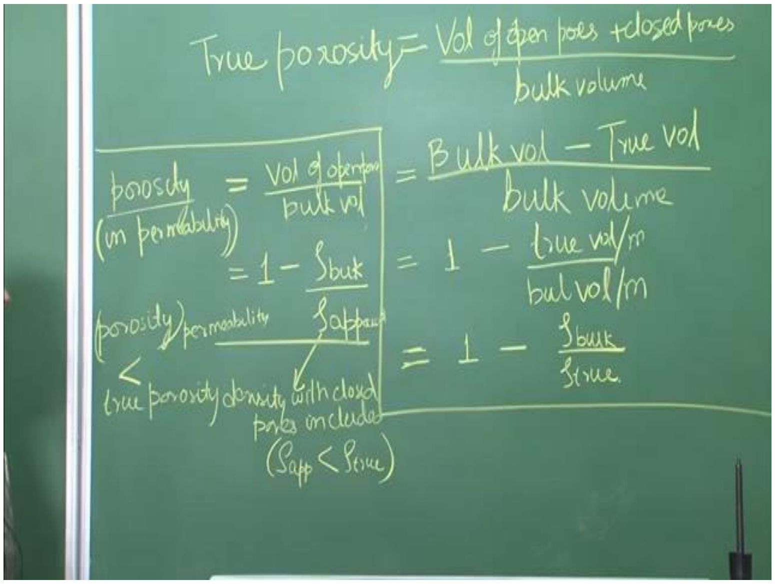

$$\text{True porosity} = \frac{\text{Vol of open pores + closed pores}}{\text{bulk volume}} = \frac{\text{Bulk vol} - \text{True vol}}{\text{bulk volume}} = 1 - \frac{\text{true vol}/m}{\text{bul vol}/m} = 1 - \frac{\rho_{bulk}}{\rho_{true}}$$

$$\underset{\text{(in permeability)}}{\text{porosity}} = \frac{\text{Vol of open pores}}{\text{bulk vol}} = 1 - \frac{\rho_{bulk}}{\rho_{apparent}}$$

$(\text{porosity})_{\text{permeability}}$ $<$ true porosity

density with closed pores included $(\rho_{app} < \rho_{true})$

So the true porosity can be written as, so we are looking at the relative point fraction so it is volume of open pores plus closed pores by the bulk volume and okay so, what will be the bulk volume over here? So the bulk volume would be, if you have taken a whole thing including the volume of the closed pore or volume of the open pore, so that would have been your bulk volume. So in ter point schematically or uh graphically what does the true porosity represent? It is the fraction of volume taken by these pores divided by the whole volume.

So that is your true porosity. And in terms of the true volume and apparent volume we can write it like this, this will become bulk volume minus true volume divided by bulk volume, and therefore you can write it as one minus true volume by bulk volume and if you divide by mass on both sides, then this becomes one over row, one over true row, this becomes one over point row bulk and therefore you can write it as, so this row will be in a numerator and this will become row bulk by row true.

Now this is your true porosity, but like I said this is, this true porosity is also including your open pores and closed pores but when we are using the permeability method the gas permeability method we know that we are not using the closed pores. And therefore, the porosity that we are using in our permeability method that will be a little bit different, and you can say that this porosity, so let me point classify or refine it here that in permeability.

So this will now we can say is equal to, so we can take away the closed pore we will have only the open pores by bulk volume and if you rearrange it you can see that it will become 1- row bulk by what is called as row apparent, now what is this row apparent? It is the density when you are calculating point when you are taking into taking closed pores also into account density with closed pores included. And therefore this point row apparent like I said we are taking, this is your apparent value, so you are taking this as the volume, so the volume amount is larger than what it should be in the true volume.

And therefore the density will come out to be smaller. So this row apparent is smaller than row true because the denominator that we are taking point which is mass by volume in over here this is larger, in over here this is smaller and therefore this becomes a smaller quantity. And that means overall this porosity that we are calculating if you look at the whole term over here and compare it with the true porosity, this is a larger value. So this porosity if we were to write porosity, point sorry let me refine that, so this porosity is smaller because this is of course only taking open pores and not the closed pores.

So this porosity which is from point which we have used for permeability is smaller than true porosity. So overall it gives you an understanding of what are the different kinds of pores and which are the pores being taken into account when we are talking about permeability. And which are the pores you should ideally be taking into account when we are talking about point some actual surface area measurement method. So with that you can say that there is going to be some difference in the surface area that you calculate using gas permeability method and say BET method.

Point but however, having said that it will still give you a good point you can say relation between the porosity and the sur point the point and the surface area and that can be related to find the surface area that we have been trying to do. So hence this method will normally underestimate se so what like I said that there will be difference in the porosity, therefore this method will normally underestimate the surface area with respect to BET method.

However, you can also use this to find out that difference, that difference will be proportional to the surface area related to closed pores. So that gives you also a tool to basically differentiate between open pores and closed pores. Because one method is giving you the surface area because of both closed pores and the open pores which is BET and the gas permeability method is giving you only the open pores and therefore and you compare the two result, you can also say what is the surface area that is related to closed pores.

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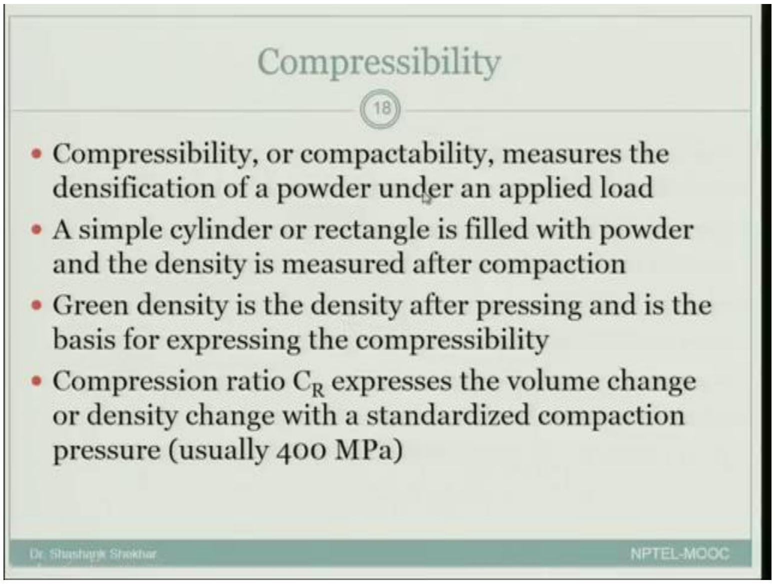

Compressibility

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- Compressibility, or compactability, measures the densification of a powder under an applied load
- A simple cylinder or rectangle is filled with powder and the density is measured after compaction
- Green density is the density after pressing and is the basis for expressing the compressibility
- Compression ratio C_R expresses the volume change or density change with a standardized compaction pressure (usually 400 MPa)

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So that is another method that we looked at gas permeability method. So these are the most of the techniques that are used for point characterizing powder. However, there are some parameters point that point are useful to characterize powder, and one of them is compressibility because we are talking about powder processing where powder will we compressed. So let us look at what is this compressibility. Compressibility or compactability meaning how much you are able to compact measures the densification of powder under an applied load. So it tells you how much this powder can be compressed or compacted.

A simple cylinder or rectangle is filled with powder and the density is measured after compaction. Now you point you know that we already are aware of the true density, but what we are measuring over here is not the true density, we will be measuring what is called as tab density or the green compact density we are just applying a pressure and measuring how much it can compact or you can even find it in relation with the true density. So you can say it is 60 percent of 0.6 of the true density.

So that will be, that is the usual measure of the compressibility or compactabiltiy what is the fraction of this compressed powder with respect to true density. So green density is the density after pressing, so this is the after pressing you get green density and even if you, even before you have pressed it, there will be some density that you can call as the tab density. So you first get the tap density point which includes not only the open pores and the closed pores but also some area, some volume in between the particles.

Then you compress it and you get what is called as the green density. And then you also define a term like which is called compression ratio, which expresses the volume change or density change with a standardized compaction pressure. So you can take different powder, let say copper and point iron powders and put standard pressure which is 400 mega pascal usually and compress them, and find out what is the change in volume or density change, relative density change and that will be called as the compressibility.

And from there you can say whether copper is better suited for powder processing or iron powders are better suited for powder processing. So this gives you an understanding of which powder is better suited for point compressing. So this is a very important parameter. Another important parameter when we are talking about powder is agglomeration. Now we already know that particles we are talking about are very small, they are point very small in size. Now whenever the particles sizes are very small, there will be a tendency to agglomerate. Why would there be a tendency to agglomerate? Let us see.

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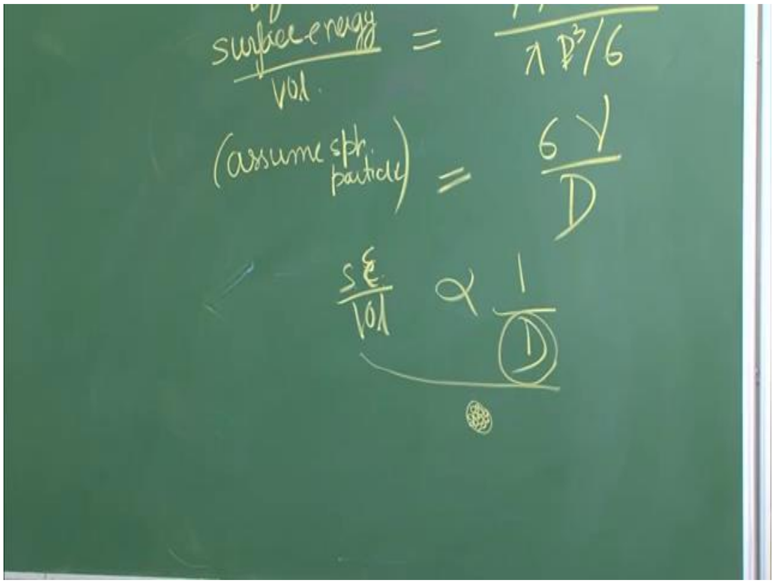

If you are talking about point powders or the size of few macrons this becomes even more important. Let us look at surface energy per unit volume. For a particle of given diameter d, now if the surface energy per unit area is gamma, then surface energy for a particle, a spherical particle we are assuming, you can do a similar exercise for let us say a cubical particle and find out what will be the surface energy per unit volume how will it relate with a given length if it is a cube let us say l is the length. In point sphere d is one of the characteristic length.

So the energy will be pi d square, this is the total surface energy for our spherical particle of d, particle size d and the volume is pi d cube by six, therefore this comes out to be, so what do you see surface energy is, surface energy per unit volume to be precise is inversely proportional to d. Now you know that energy will always for a system energy always tries to reduce. And therefore what will happen is that how will I try to reduce this surface energy per unit volume term? By increasing d. So the pati, the system will try to have larger d on an average so that the surface energy per unit volume is lower, but the particle that you are producing, that is depended, the size of the particle is depended on the process parameters.

And therefore that cannot be changed. So what the particle does is put together or the system not the particle but the system does is get a lot of particles together, so the particles have the tendency to come together and because of the reduction in surface energy which is gam point , which is proportional to six gamma by the, they will come together, and therefore even though the small particle size was a small, very small size but they can come together and forma agglomerate whose affective diameter will be much larger than one particle size diameter.

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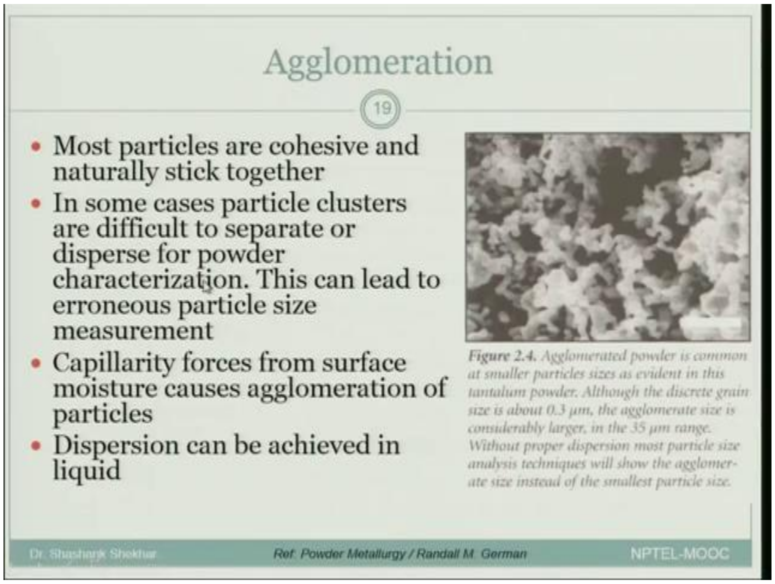

Agglomeration

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- Most particles are cohesive and naturally stick together
- In some cases particle clusters are difficult to separate or disperse for powder characterization. This can lead to erroneous particle size measurement
- Capillarity forces from surface moisture causes agglomeration of particles
- Dispersion can be achieved in liquid

Figure 2.4. *Agglomerated powder is common at smaller particles sizes as evident in this tantalum powder. Although the discrete grain size is about 0.3 μm, the agglomerate size is considerably larger, in the 35 μm range. Without proper dispersion most particle size analysis techniques will show the agglomerate size instead of the smallest particle size.*

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So that is the driving force behind agglomeration, that is the surf, reduction in the surface energy. So let us look at the slide here, the most particles are cohesive and naturally stick together and why are cohesive? Again the driving force is the reduction in the surface energy. In some cases, particle clusters are difficult to separate or disperse for powder characterization. So sometimes it may happen that the particles have point agglomerated and come together so much that it is not even possible to separate them out.

This can lead to a erroneous particle size measurement and in that case if you are doing the measurement the surface energy, for example say, let say the total surface area, that has reduced because that was the driving force and therefore the particles size measurement if you do will be erroneous. So you have to be careful to ensure that there is no agglomeration when you are calculating the particle size. Capillarity forces from surface moisture causes agglomeration of particles. So you can call it capillarity forces or the reduction in the surface energy term which leads to this point reduction in the surface energy.

Compression can be point sorry dispersion can achieved in liquid, so let us you have achieved point or you have obtained agglomerated particle. One of the best way to disperse them is to put it in a liquid. Now in the liquid the surface energy term changes and because of that you can get dispersion and therefore you will be able to have particle size, particles of their natural size and then you can do the point characterization and that is why you remember in most of the techniques that I point showed you, the particles where actually in some of the solution, when they are in some of the solution then they do not stick to each other and they remain separated and then you can calculate the actual particle size.

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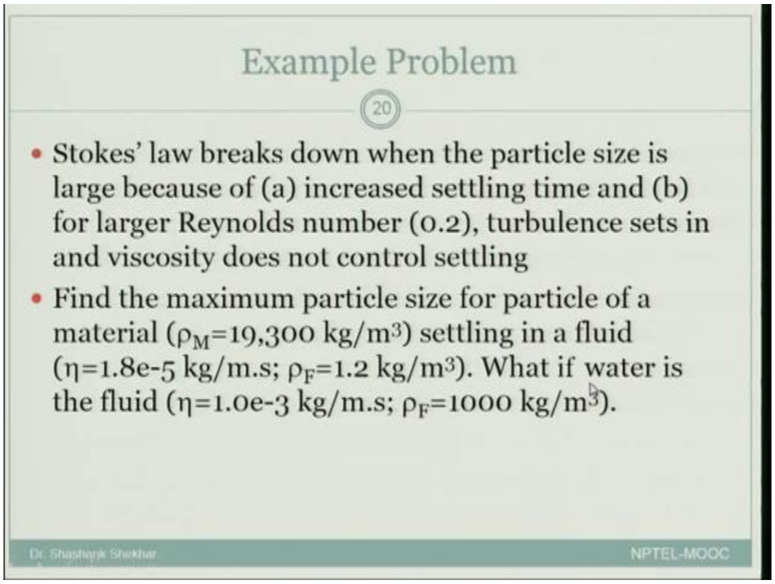

Example Problem

- Stokes' law breaks down when the particle size is large because of (a) increased settling time and (b) for larger Reynolds number (0.2), turbulence sets in and viscosity does not control settling
- Find the maximum particle size for particle of a material (ρ_M=19,300 kg/m^3) settling in a fluid (η=1.8e-5 kg/m.s; ρ_F=1.2 kg/m^3). What if water is the fluid (η=1.0e-3 kg/m.s; ρ_F=1000 kg/m^3).

So at this point, point we are coming to end to the characterization of point the powder particles, so we I promise that we will take a look at example problem based on this particularly to show the that there is an upper size that can be characterized using the sedimentation method, you remember sedimentation method was based on stokes law which says that a fall in particle reaches its point terminal velocity and the terminal, from the terminal velocity you can calculate the distance and the time and therefore you can say what is the approximate particle size.

There was a lower limit, and that lower limit was because if the particles are two small they will remain suspended in the point fluid medium whether it is liquid or gas it will remain, it will remain suspended and therefore it will never reach and therefore you cannot calculate the particle size. On the other hand, on the other extreme, there is also a upper limit to the particles size and that is determine because with point by the Reynolds number. Now when the particles size becomes larger and larger, the turbulence sets in, and when turbulence sets in, Reynolds number increases.

That happens at approximately point Reynolds number of 0.2. So let us say the Reynolds number is given as 0.2, you are given to find the maximum particles size for particles of a material who's row m is given and point it is settling in a fluid, so the fluid density, fluid point viscosity is given, fluid density is also given and then you have to do the same calculation for point another fluid, the difference being that this time the viscosity is very very different and also the density is very very different. So we will see how these parameters will make a difference to the maximum particles size. So let us point try this point problem, so now remember what we are trying to do? We are trying to get maximum particles size that can be characterized using sedimentation.

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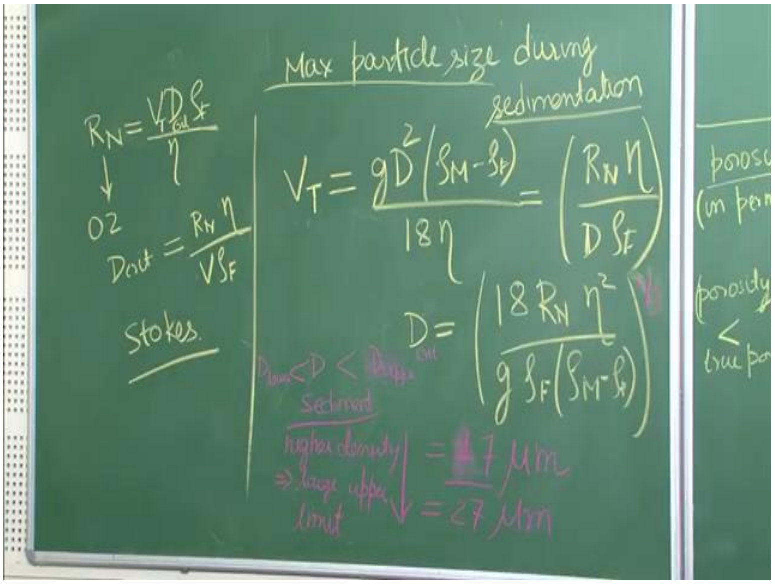

Now here we need another equation that we have not discussed in our regular class and that is the relation of Reynolds number with velocity diameter of the particle fluid density and its viscosity. Now we said that Reynolds number is point given as 0.2 or it is that Reynolds number where turbulence sets in and point we cannot apply the stokes law. So let, so we are given that this is 0.2 and this is proportional to d. So in general you would want the particle size d to be smaller than this critical size. So let us say we are finding that critical size and the critical and the diameter of the real particles should be smaller than this.

So our d critical should be, will be equal, will be equal to Rn times V row f and we also have the diameter from the other equation where we had point , where we used the stokes law. So we want to say what will be the d critical point that we obtained from here and put that value into stokes law. So for that what we will do is, we will calculate the terminal velocity which is over here, so let us say this is happening at some terminal velocity Vt for this critical diameter d. So the Vt will equate V that point critical value of V that is obtained from Rn and the critical value of V point , not critical but the terminal velocity that we obtained from stokes law.

So we put them together and we get, okay so let us understand what we are trying to say here, for a particular size, critical size diameter d, there is this velocity v. So the velocity V, this is the velocity Vt, now this velocity Vt is the largest point that the particles should have, otherwise it will start to get into the turbulent mode. And we are also saying that this is the same velocity or the particle size is such that we are getting the terminal velocity for this particular point particle.

Therefore, the t velocity, terminal velocity is same and therefore we have equated it over here and from here we will get d cube, sorry this in not one by three yet, so this is d cube and this is equal to this and therefore if you want to find d, you will take away three from here and put one over three over here. So this will give you that d critical, critical diameter if your size is less than this, then velocity Vt will remain below the terminal base velocity will remain below a veloc a value at which turbulence will not set in and even though it reaches terminal velocity which most particles will reach, so you would we able to use stokes law.

So we are equating the velocity V over here and from there we are getting the d critical, if you put in these values, you would see that this comes out to be 27 micro meter. So you cannot put particles or you cannot characterize particles which have values larger than 27 micro meter. And not only that, we also have you remember from our earlier discussion when we were discussing about sedimentation that there is a lower limit and so there is d should be less than 27 or not 27 let me say some d upper limit and it should be greater than some d lower limit for sedimentation.

Having said that now we second part of the question is to find out the d critical when we change the value of the liquid. So the if you see compare the values, let us get to the slide to see what is the density comparison. So the first fluid has density of 1.2 kg per meter cube and compare with the other fluids which is water, the density is 1000 kg per meter cube, so it is three orders of magnitude larger. On the other hand viscosity is very very low for the first fluid, it is of the order 1.8 into 10 to the power -5 and for the other liquid it is, which is water for that point viscosity is 1 into 10 to the power -3.

So this is again two orders of magnitude larger. So viscosity is larger and density is larger. How does that make a difference? And when you solve this, what you would see is that, this will come point 17 micro meter and this will increase to 27 micro meter. So point basically when you go from lower density liquid to higher density liquid, you are able to increase upper limit. So let us write it down what we have, what has happened when you use a higher density implies larger upper limit.

So just point one correction over here, V will get 17 micro meter for the first fluid and 27 micro meter for the twenty, for the second fluid which is water. So when you increase the density, so this was a thousand kg per meter cube fluid which is water and this was just 1.2 kg per meter cube. So when you increase the density, our particle size limit has increased. But what is again interesting is that the magnitude of increase here is very small, you see we have increased the density three orders of magnitude, viscosity two orders of magnitude, but the size has increased by only 50 percent.

So that is very a small increase, although it will be very significant because your lower limit may probably be of the order of 1 or 2micron. So we are talking about 2 raise to 17 and then we go from 2 to 27. So we have to use a proper liquid depending on what is the particle size and with proper viscosity to be able to characterize powders. So this is the example that point gives us an understanding or feel of why there is a lower limit as well as an upper limit when we are using sedimentation based on stokes law.

So well that is the end of our powder characterization. In the next lecture we will start with point next the topic which is order fabrication or powder manufacturing. So we will be discussing about different techniques which are used for making the powders. So thank you and see you in next class.