Particle Characterization
Prof. Dr. R. Nagarajan
Department of Chemical Engineering
Indian Institute of Technology, Madras

Module No. # 03
Lecture No. # 10
Morphological Characterization: Particle size distributions

Welcome to the tenth lecture in our particle characterization course. In the previous lecture, we continued our discussion of a particle size characterization. And the key point that we were making in the previous lecture is, that given the uncertainties involved in estimating the sizes of particles using light scattering techniques, it is very important to use a particle counter or size analyzer, which is properly calibrated as well as correlated against a traceable standard, and it is important to keep track of not only drifts in the actual functioning of the equipment, but also the procedure by which the particle counts are size analysis are done.

The sources of error in the procedure can actually be much greater than the sources of error; for example, in the laser operation itself. So, it is very important to understand exactly how sample preparation is to be done and to be able to do it in a repeatable and reproducible manner in every time.

The other point that we just started discussing in the last lecture is the fact that if you look at particle populations in general, they are naturally what we call poly dispersed. In other words, we see a spectrum of particles sizes - it is very rare that we find a particle population that contains only one size unless it is a specially prepared standard. For example, the poly styrene latex or PSL standards that we talked about in the last lecture which are used for calibrating particle counter or a particle size analyzer are deliberately prepared in various size ranges in order for us to be able to carry out this calibrations exercise, but in general, if I were to just sample particles that have accumulated on the surface or particles that are present in this air around us, you will see a variety of particle sizes.

In fact, in one of the earlier lectures, I sketch the typical prevailing particle distribution in an outdoor environment and in an indoor environment and we saw that both of them have essentially a tri-model characteristic.

So, given that, the fact that we deal mostly with particle size distributions means that our method of size measurement should also enable us to characterize particles not only of one size but sizes ranging from a minimum to a maximum size.

And particle counters are designed to do exactly that. If you look at particle size distributions that are prevailing in nature, they can broadly be classified in to two categories - one is called the normal distribution and the other is log normal distribution.

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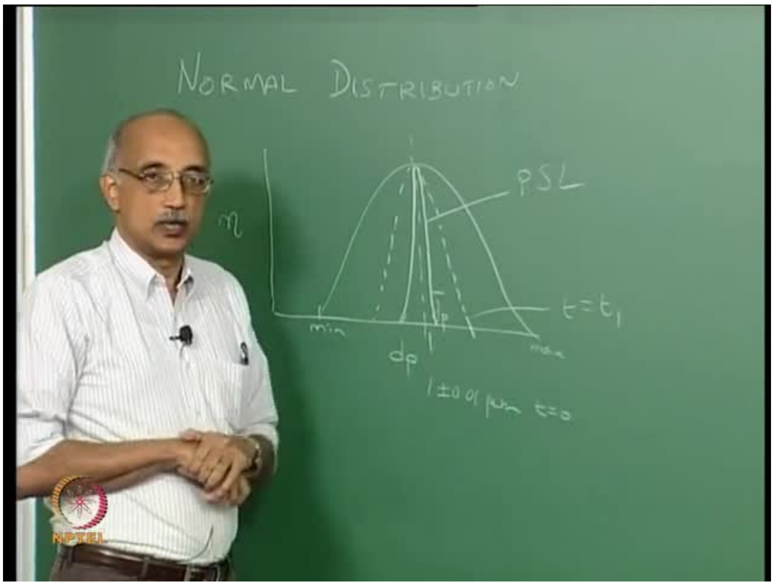

So, these are the two most fundamental particle size distributions that you should be aware of. So, what is the difference between the two? What do we call a normal distribution?

In a normal distribution of particle size, if you plot the size of the particle on the x axis versus let say the number of particles corresponding to that size on the y axis. What you would find is a distribution that looks like this.

So, there will be a mean size corresponding to this population. There will be a minimum size; there will be a maximum size and there will be a standard deviation or variability value which describes the spread in the population. This is call a normal distribution because essentially it describes a Gaussian behavior.

So, you can apply standard statistical techniques. I am sure you have studied how to calculate the mean of a distribution, the standard deviation of a distribution and so on.

All those formulae are really only applicable to such normal distributions; so, they can be applied in this case, but how do you get such a distribution in nature? The way you get normal distribution in nature is by starting out with a distribution that is much tighter.

For example, let us say that you have prepared PSL standards that are, let us say this is 1 micron, that are closely centered around the 1 micron size. You have done the synthesis in a lab, you have done size enlargement or size reduction. To achieve a population of the PSL particles that are, let say 1 plus or minus point 0 1 microns in size very tightly clustered around this 1 micron size and let us say that this is your calibration standard that you are going to use to calibrate your particle counter or particle size analyzer.

So, what happens, if you take a bowl or a beaker containing these calibration standards and just let it stay on the shelf for some period of time, suppose you look at this same distribution after a month of shelf storage, what is the population going to look like? It is going to show science of broadening. So, this may be at time 0; this will be at t equal some t 1. Why does broadening of the distribution happen? Because of additive and subtractive effects. So, you take an initially mono dispersed population and you allow addition and subtraction phenomena to happen and you start seeing a broadening of the spread in the data.

So, eventually, over a period of time, this may be 1 months, 2 months, 3 months, 4 months. The distribution will keep getting wider and wider.

Now, why does that happen? In this particular case, it happens because the growth phenomenon is driven by agglomeration particles that are in suspension tend to join each other and grow larger in size.

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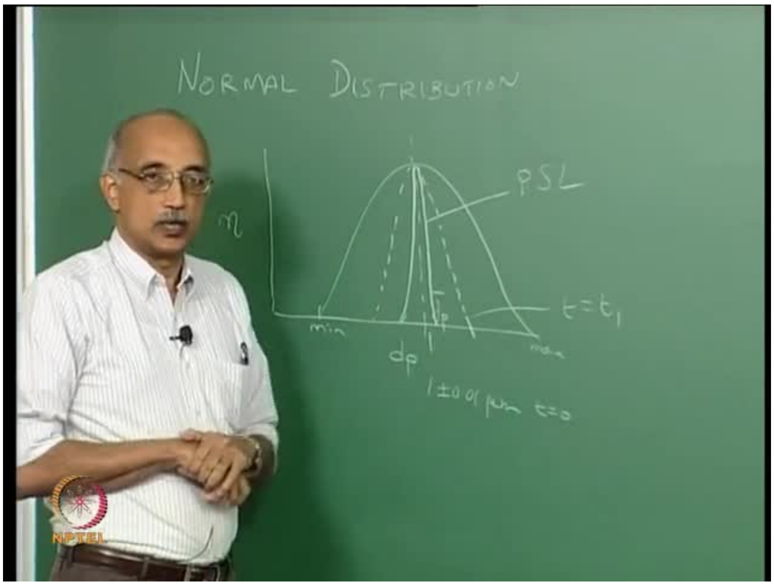

So, that is shifts the maximum sizes upwards as you start forming these agglomerates. Now, why do you see a broadening in this direction? That is predominantly because of settling as well as interception phenomena.

So, if you have a beaker, many of the large particles will have a tendency to slowly sediment and essentially form a powdery layer on the bottom of the beaker.

So, unless you keep re-suspending these particles, you are continuously loosing large particles and also some of the particles are drifting to the sides and sticking to the walls of the beaker and so on.

So, in effect it shifts the population towards the left. So, the combination of particle size gain and size loss due to these phenomena will result in a widening of this distribution.

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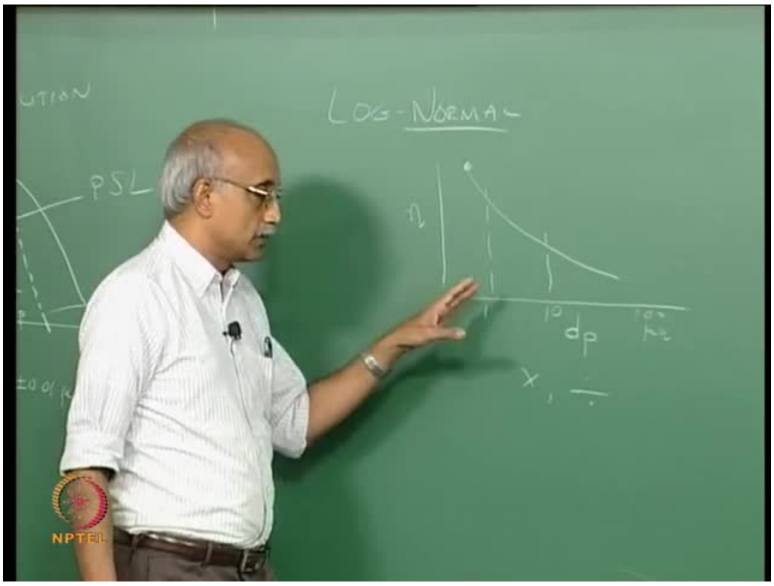

So, this is what we call a normal distribution, but actually when you compare this to the next distribution, we are going to talk about which is the log-normal distribution. This is less likely to happen in nature.

This is more likely to happen under control conditions where you have a pre-characterized population of sizes to begin with and something happens to alter that size distribution.

On the other hand, the log normal distribution can truly occur in nature, and how does it happen? Again if you plot particle size versus count, as we have seen before in the lowest size range, nucleation and condensation are significant phenomena.

Now, when nucleation and condensation happen, you will get a fairly large number of particles, because a nucleated aero salts or particles are very fine in size. The first nuclei that form actually are in the angstrom range but we do not really consider them as particles until they grow too roughly at least 0.1 nanometers in size, and so, the first agglomerates are form are very fine, the first nuclei that form, and then, they will eventually grow to a size that is still very low on an absolute size scale but larger than vapours or vapour molecules.

So, this phenomenon is one that really creates literally 1000’s and 10’s and 1000’s of particles in this very fine size range. Now, what is going to happen to these particles?

Some of them will (( )) and grow and they will start producing particles that are in a larger size range, and so, you will see particles eventually start to appear in the larger sizes but the number will be smaller compared to the very fine sizes, and in fact, in the log normal distribution, the kind of trend that you see is one where there is a significant enrichment of particle counts in the finer sizes compared to the larger sizes.

So, another way of saying that in nature - if you can see particles that are, let us say we can see 10 particles or 100 microns in size, there is at least 1000 particles that are 10 microns inside that you do not see.

And there are at least, you know, 10,000 particles in the 1 micron size which you do not see. So, in nature, for every coarse particle or large particle that you see, there is an associated number of finer particles which are much greater in quantity and the log normal distribution essentially reflects that reality.

So, how does this happen? As I said earlier, normal distribution happens because of plus minus phenomena additive and subtractive.

Log normal distributions happen because of multiplicative and divisive phenomena. So, what do we mean that? What is the typical multiplicative phenomenon? Well, actually nucleation and condensation are considered multiplicative phenomena because they are not additive in nature. Essentially you can take a single droplet or a single large concentration of vapour molecules in the gas phase which can nucleate and condense to give a certain numbers of particles which are not necessarily additively or subtractively related to the initial population of vapour molecules in a gas phase.

What is a typical divisive phenomenon? Aberration; when you take a surface and you upgraded with another surface, the number of particles you generate are going to be against fairly large in count and they are happening essentially because you are taking a previously continuous surface and you are upgrading in to form finer particles.

So, you essentially taking a large mass or volume of material and dividing it into finer fragments. So, these are the two primary contributing phenomena to the formation of a log-normal population, and again if you look at nature, let us say the environment, the atmosphere, many of the particles that we see suspended in air are formed because of these two mechanisms - nucleation and condensation - followed by growth and mechanisms of aberration or entrainment that happens in a divisive manner, and so, if you measure particles, for example, in this room, in air, you will typically find this type of distribution.

And of course, if you now look for particles on a surface like this table and you look at the distribution, again it is very likely to follow the same distribution because the source of particles that are depositing on the surface is the air that surrounding it. So, the distribution of sizes on a surface is likely to be very similar to the distribution of particle sizes in the surrounding fluid.

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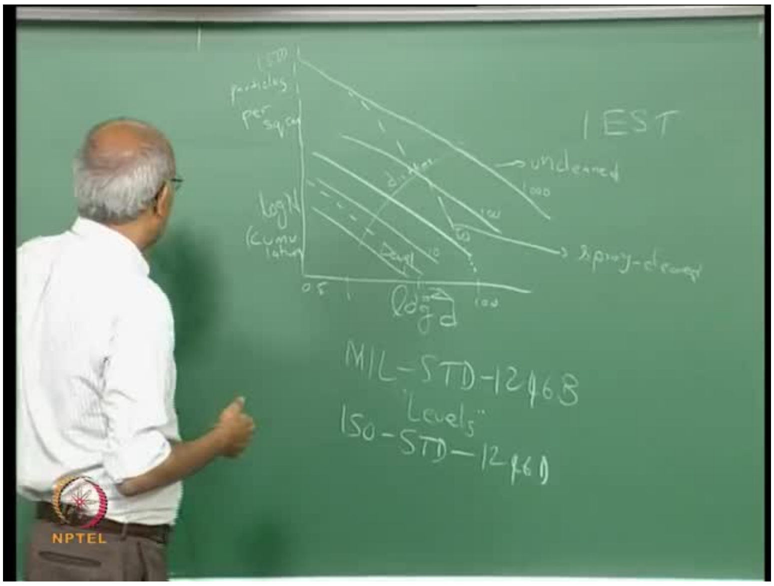

So, if a take a surface and let say I plot logarithm of size squared versus logarithm of count of particles that are larger than that size, so, this is a cumulative count now. Will look at the difference between cumulative and differential counts later in this lecture. It turns out that they all follow straight lines.

If you plot it as log log squared on the two axis and this is called a log normal distribution. All particle population that show log normal distribution will show a linear relationship between square of the logarithm of size to logarithm of the cumulative count corresponding to that size and it turns out that for depending on how dirty or clean the environment is this count absolute, count can vary but the slope remains the same.

So, you can have a family of these parallel lines where each of these lines obviously as you go up, it is getting dirtier. In other words, for the same size, you have a lot more particles as you go up in this curve, and so, each of these lines has been designated as a level.

So, this would be a level 1, this may be a level 10, this may be a level 50, 100, 1000, (Refer Slide time: 15:59) and so on, and these levels are actually reflective of how clean a surface is.

So, for example, if I say that this table is a level 1000 surface, what it means is that if I plot the particle counts verses the size, it will lie along this line where the intercept on the y axis will correspond to 1000 particles, and this line starts for this purpose at 0.5 microns, 1000 particles per square centimeter.

So, the definition of a level 1000 surface is that it contains 1000 particles that are half a micron or larger per square centimeter of the surface.

In fact, there is a MIL-standard that was written many years ago; its call the MIL-standard 1246 B; it is actually developed by aerospace industry in the US to describe how clean space craft surfaces needed to be and they define these levels.

So, these levels that we talked about are all related to this MIL-standard 1246 B. Now, more recently these have been adopted as ISO-standards and the latest designation as ISO-standard 1246 D.

Essentially, there is an organization called the institute of environmental sciences and technology, IEST, which has been given responsibility for taking these existing standards from various industries – aerospace, semiconductor and so on, and merging them into one universal standard.

So, these ISO standards are an essentially a global standard that are now being used to particularly in this case describe particle populations on a surface.

So, if I have to go to somebody and say that only surfaces and ISO standard 1246 D level 1000, they will immediately know what I am talking about, and since the slope is fixed, once I say what the count is at 0.5 microns, you can actually calculate what the corresponding counts would be at 1 micron, 10 microns and 100 microns and so on.

And again given the slope of the curve, if I have 1000 particles that are half a micron and larger, the probability is by the time I get to 100 microns, the number will be very small.

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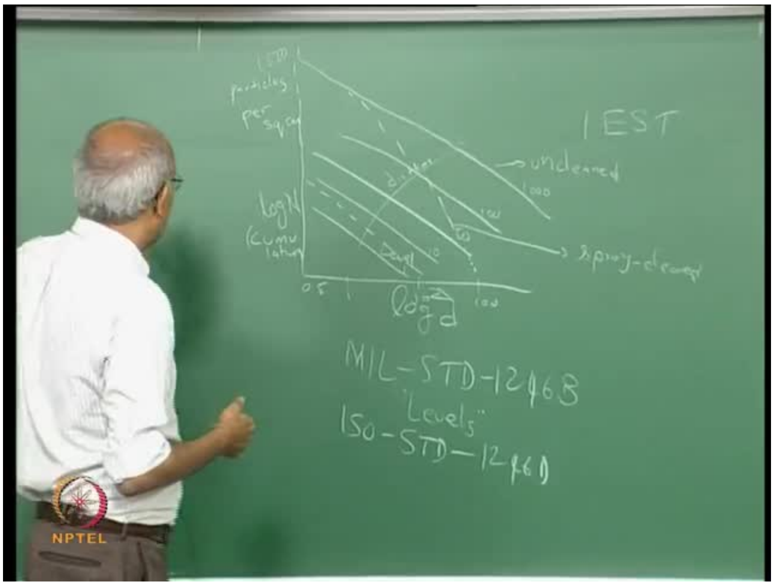

Because again, when we talk about naturally occurring contamination on a surface, there is a linear downward slope when you plot it as a log log square. So, this is an interesting behavior, and by the way if you take this surface, an actually apply a process to it.

For example, if I clean it, then this distribution will not apply any more, because as soon as you do some thing to the surface to disturb the particle population, you are going to alter the distribution.

So, this is essentially for a virgin surface on which particles are accumulating naturally. As we will see later in the course there are techniques, there are very effective at removing particles from surfaces. For example, if we have a dirty surface here and I spray water on it, I will remove a majority of the material on it.

However, when we spray water, we will preferentially remove larger particles. So, if I take this population that is present and I spray clean the surfaces, what is going to happen to the particle size distribution?

It is going to be even more vertical. I am going to see a slope like this. So, if this is an un-cleaned level 1000 surface, then this is for a spray-cleaned surface which was a level 1000 to begin with. So, essentially when you use a technique that based on shear forces, it can remove large particles very effectively but it leaves these finer particles undisturbed.

So, the counts at half micron level may remain unchanged even after you clean it with the water or whatever, but there will be a drastic reduction in the particle count in the larger sizes.

There are other techniques will talk about which are much more effective in removing finer particles, such as, ultrasonic cleaning or mega sonic cleaning. If I take this same surface and instead of doing spray cleaning, I did ultrasonic cleaning, then I would expect to see very different behavior even the counts that half a micron would be rapidly reduced and I now might see a trend that looks like this.

So, these MIL-standard or I ISO-standard 1246 levels of surface particle counts can be used very effectively to assess initial particle population on a surface. They can also be used to assess the effectiveness of various cleaning processes that are used to make the surfaces cleaner for our use.

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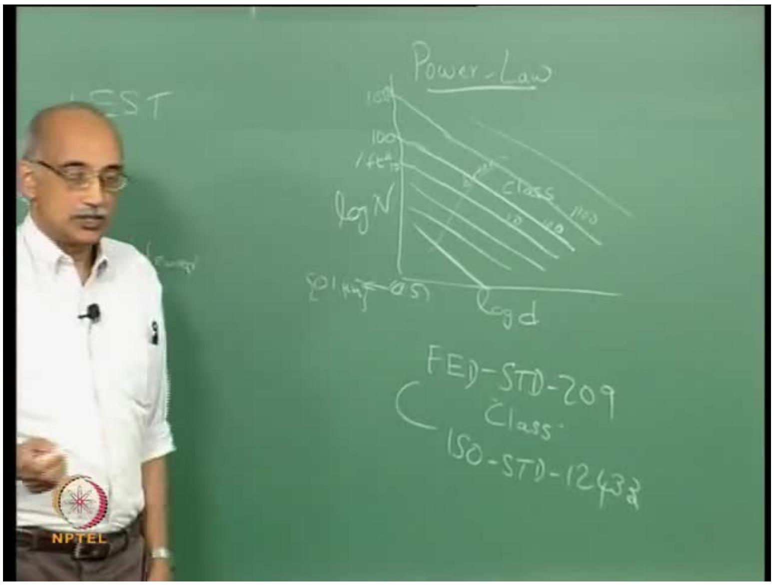

Now, we have talked about two types of distributions - normal distribution and the log normal distribution. The third type of distribution that is very important to be aware of, that is what we call power-law distribution.

A power law distribution is one that applies to environments that are actively controlled with respective particles that are present in this environment. A classic example is a clean room where you have filters that are design to remove particles, remove and retain particles from the air that is entering the clean room.

So, what are you doing in that case? You are essentially truncating the particle population. Well design filter will remove particles in a very wide size range, ranging from millimeters to micrometers and allow only very very few particles to enter the controlled environment. So, when we look at the particle size distribution in such a clean room or controlled environment, and again we plot. In this case, log of the diameter versus log of the particle count. We find that we see a similar straight line and these are called class lines.

So, just like in a case of the log normal distribution. In the power log case also you will see a family of these straight lines, and just as in that case, as you go up, it is a dirtier environment, and as in the previous case, the different classes are labeled based on their intersection with the 0.5 micron line.

So, for example, if the cumulative count of particles, in this case per cubic feet, for particles larger than half a micron is 100, then this is called a class 100 clean room. So, a class 1000 clean room will then have 1000 particles per cubic foot which are half a micron or larger, and a class 10 clean room will have 10 particles per cubic foot which are half a micron or larger.

So, just like in the case of the level, it is a reflection of the celandines of a surface. Similarly, these classes or a reflection of particles that are present in the ambient environment, and just like there is a standard that applies to particles on a surface, the clean room standards where actually developed as a FED standard and the original name of it was FED standard 209.

So, the federal standard 209 which was again developed in the aerospace industry specified classes of clean rooms. So, this standard specified that a class 100 clean room can only have a 100 particles per cubic foot that is half a micron and larger.

So, this enable various people that we are manufacturing product in clean rooms to compare there clean room facilities, to specify their clean room facilities, to monitor their facilities and to verify conformance to certain specifications, and just like in the other case, this also now has been picked up by ISO and the corresponding ISO standard is ISO standard 12433 which essentially did two things - one is that it took this clean room classes that were based on FPS units and it converted them to metric units.

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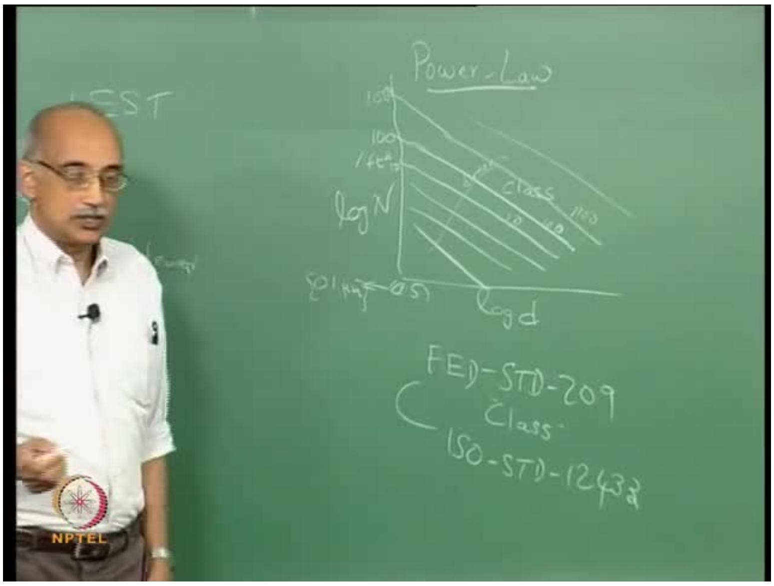

So that they are more universally usable and also it extended the definition of the minimum size from 0.5 to now 0.1 micron, because as manufacturing tolerance as become tighter and tighter, the manufacturing clean rooms needed to become cleaner and cleaner.

So, the industry has been driving the size downwards, and so, accordingly, these classes also had to be refine so that they are applicable to cleaner manufacturing environments than what people were using decades ago.

So, the combination of these clean room classes and surface contamination levels is used very widely in the high technology manufacturing industry such as, semiconductors - to specify and control how clean the manufacturing environment is from a particle view point and also how clean the product surfaces are again from a particle view point.

So, these three distributions - the normal distribution, the power law and the log normal distribution are all very relevant in a practical sense and they must really be understood and appreciated as we go forward because they gave a frames of reference.

We will look at various types of size distributions, but virtually all of them will fit into one of these three predominantly and undisturbed particle population is likely to follow either a normal distribution or a log normal distribution, whereas, a truncated or filtered population is likely to follow the power law distribution model.

Now that we have described certain classes of particle size distribution, there are commonly encountered. Let us take a closer look at how we actually obtain particle size distributions when we use a particle counter.

Now, the data from a particle counter is reported either in tabular form or in graphical form or both. So, there are some key aspects of how these data reported that we need to be aware of.

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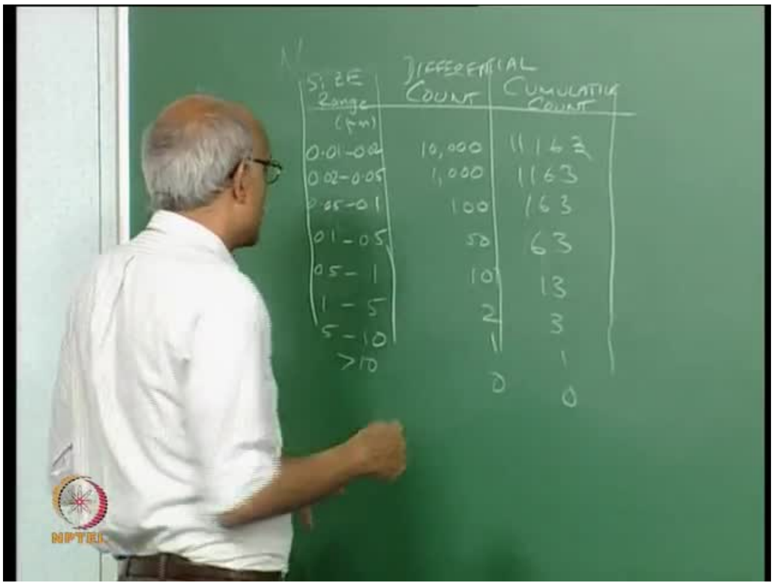

So, when we take a sample and we analyze it using a particle counter, that type of data that you will see immediately would be in a tabular form. So, the first column will have size range, let say in microns, the second column will have differential count, and third will have cumulative count.

And particle counters can have anything from 6 to 8 to 256 channels depending on the granularity of the data that you are interested in. So, the first channel may be counting particles that are, let say in the 0.01 to 0.02 micron size range or 10 nanometers to 20 nanometer size range, and let say that you are looking at a log normal population so that there is a large number of particles in the final ranges. Then you might find that in this particular size range, you have 10,000 particles.

The next channel may be from 0.02 to 0.05 microns, and you might find that in this size channel, you have 1000 particles. The next channel may be from 0.05 to 0.1, and you may have, let say 100 particles; When 0.1 to 0.5, you have 50 particles; 0.5 to 1, you have 10 particles; 1 to 5, you have 2 particles; 5 to 10, we have 1 particle, and greater than 10 microns, you have 0 particles.

This is a very typical size distribution, you will get when you looking at a log normal population where that is a much larger number of fine particles compare to coarse particles.

So, the cumulative count essentially you had backwards, so, 0, 1, 3, 13, 63, 163, 1163, 11163. So, these are the cumulative counts larger than of all particles that are larger than a particular size. So, 10,000 is the number of particles that are in the size range of 0.01 to 0.02 microns; 11,163 is the total number of particles that are larger than 0.01 microns.

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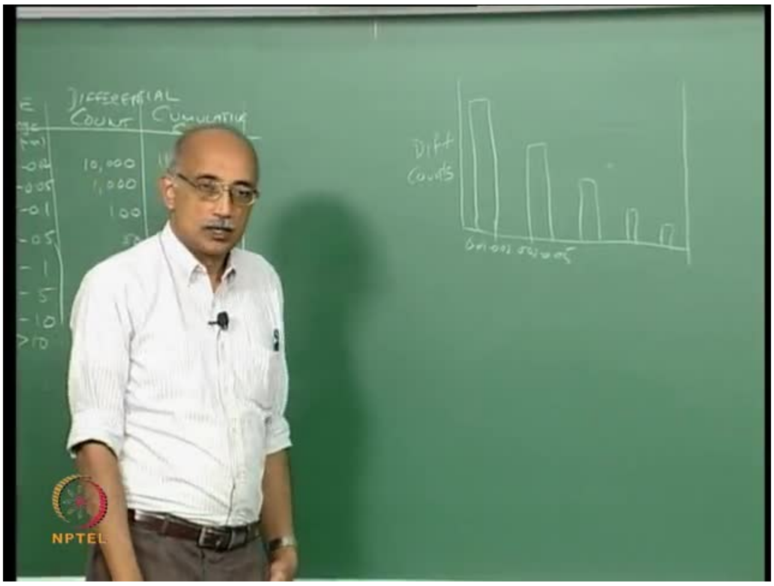

So, that is the way to interpret the data. So, what do you typically do with this kind of data? You graph it and you look at it. So, in this case, the simplest graph would be a histogram where in the first, you have to plot 0.01 to 0.02, 0.02 to 0.05 and so on.

And on this axis, you plot, let say the differential counts. So, you will have a big bar followed by a smaller bar and so on. So, this is a simple histogram representation of the data. Now, what do you do with this kind of data? Depends on your application and sometimes you are interested in, let say mean size; sometimes you are primarily interested in the differential count in a particular channel. For example, if you are manufacturing a product which you know is very sensitive to particles in a certain size range.

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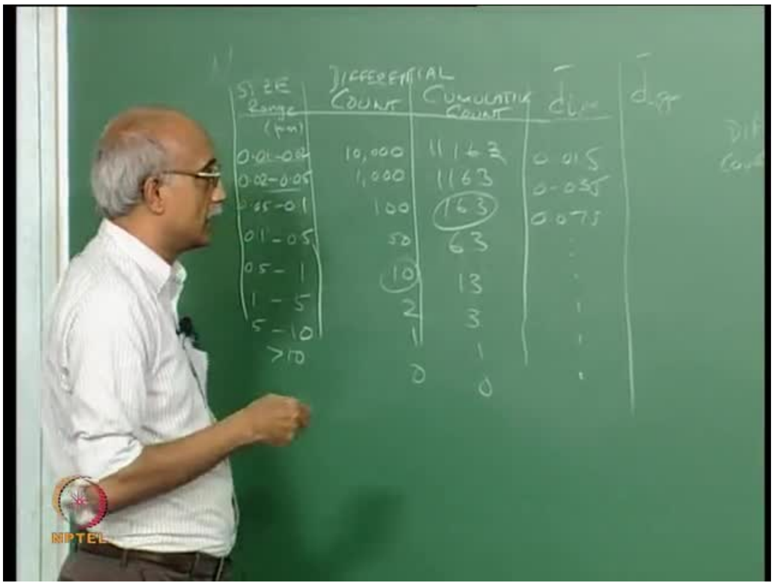

Let us say from 0.5 to 1 micron. Then, you will look at this distribution and focus primarily on the differential counts that are in that particular size range, or you might have it is product which you know a sensitive to all particles that are larger than, let say 0.05 microns. Then the key parameter that you look at will be this because this is the cumulative number of particles that are larger than 0.05 microns.

So, this is the complete set of data that are available to you, but out of these, you must choose the data that are truly relevant for your process, your application and so on.

Now, when you are graphing this data, you may also want to graph it as essentially a continuous curve, a continuous function. Now, it is always risky to do that with particle size distributions, because when you take discrete data and you converted to a continuous function, what is the assumption? The key assumption is, you know, how to fill in the gaps and that is a dangerous assumption when you are talking about particle populations.

It can depends on how dilute the sample is. If you are counting statistics are very very low, then the error in deriving a continuous function from discrete data can be very very large.

The more particles you have to look at, the more particles that are available to you to give your statistics. The more confident you will have in your ability to take discrete data and obtain a continuous function out of those.

So, that the key suggestion is when you take particle count data be very very careful in taking discrete data and converting into continuous functions. You have to apply certain statistical test to verify the validity of what you are doing, but what you would like to do then is, you would probably like to define a mean size for each channel, so, it will be mean size here is 0.015, 0.035, 0.075 and so on. For each of these channels, you can define a mean size. Now, one thing you will notice - if you look at the width of the channels, what is the thing that you that you notice first?

The width of the channel is very very small in the lower sizes and keeps getting progressively broader, because big theory is only 0.01 microns, here it is 0.03, here it is 0.05, here it is 0.4, 0.5, 4, 5 and so on.

And the reason for that is again particularly in a log normal distribution, the changes that we see in the particle counts are extremely high at smaller sizes compare to larger sizes.

So, the relative sensitivity of counts to size is much greater in the finer fragment of the population compare to the larger fragment of the population, and that is why you need to have as finer division in the small particles size scale compare to the larger particle size scale.

So, one way to plot this data would be to simply take d i bar and plotted against, let say that the cumulative count and again you will get you know some curve that will look like this. Alternatively you can, I mean this is the number mean, you may actually want to take the geometric mean, because of this problem, that mean you take a number mean, you are not giving sufficient weightage to the granularity of the widths of the various channel. So, by the taking of the geometric mean is actually much more appropriate for a MIL standard population. So, here, of course, it is basically square root of the small size and the large size corresponding to each of the size channels, so, it will be square root of 0.01 and so on. When we look at the, I mean the graphs can then again be plotted the same way.

The key thing here is how do you use this type of data. Most of the time what we are interested in it is not so much how the distribution looks but rather is how the key statistics that we can derive from it.

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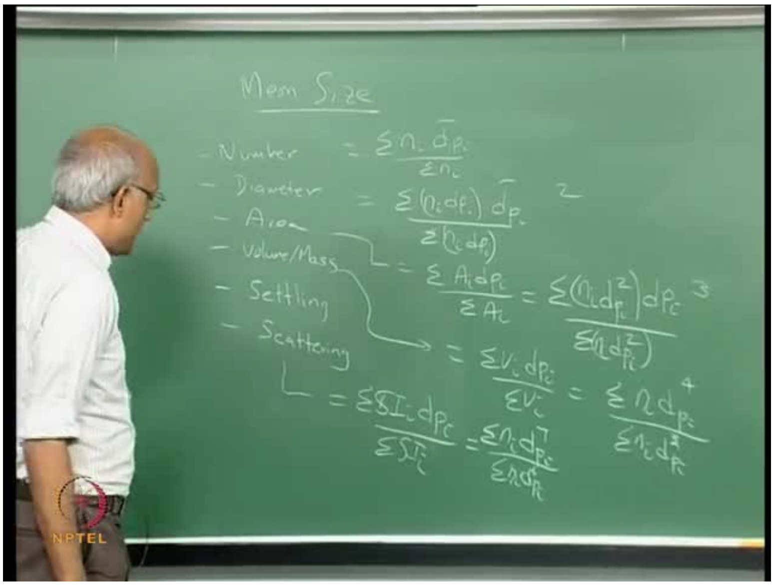

So, for a particle population, we can again think about various means, mean sizes, for example, or standard deviations. When we talk about the mean size of a particle population, it is important to realize that there is no unique mean size. It depends on how you define mean size. You can essentially take different movements of the particle population and define different means based on which movement you are considering.

So, when we talk about mean size, we can talk about a number mean, we can talk about a diameter mean, we can talk about an area mean, we can talk about a volume or mass mean diameter.

We can even talk about a settling mean diameter or a scattering mean diameter, and again you have to make a choice for your application, which is the mean diameter that is physically relevant.

For example, if you are trying to measure catalyst particles, then it is very likely that the most relevant size out of these is an area mean diameter, because, you know, for a catalyst, it is really the area that has a greatest baring on it is performance.

Whereas, let us say that you are trying to measure how mixed affects visibility. In that case, a volume mean diameter may be the most relevant because what maters is what volume of air is occupied by these droplets that are present in the environment.

Scattering mean diameter: if you are using a light scattering instrument to measure size, then the most logical size to use as an average size is a scattering mean diameter, because the instrument is estimating the size of the individual particles by using scattering as a technique.

So, in order to be consistent with the functioning of the instrument, you would probably want to use scattering mean diameter.

So, how do we define these in each of these cases? So, what is number mean? Now, very simply, I mean you can write this as summation n i over d Pi bar over summation n i.

Where, d Pi bar is the mean diameter in each of those channels that we were describing earlier. A diameter mean size would be simply summation n i d Pi times d Pi bar over summation n i d Pi, and area mean diameter, actually one way to look at this is that what you are really calculating is summation A i d Pi over summation A i. This is the definition of an area mean diameter. So, what this is in terms of particle size would be approximately summation n i d Pi squared d Pi over summation n i d Pi square. So, you know, what we are seeing is that, in this case, you are taking summation n i d Pi.

So, here, multiplying d Pi by d Pi, you are taking essentially the second moment here, you are taking the third moment, and when you start talking about the volume mean diameter which is summation V i d Pi over summation V I, then this becomes summation n i d Pi cubed multiply by this d Pi. So, it is actually a d Pi to the power 4 divided by summation n i d Pi cubed and so on.

And similarly, finally when we come to a scattering mean diameter, this will be summation, let us call it as scattering intensity i d Pi over summation scattering intensity i which is equal to summation n i d Pi. Since the scattering intensity varies as d P to the power 6.

So, this will essentially become n i d Pi to the power 7 divided by summation n i d Pi to the power 6. As you can imagine, all these diameters can be substantially different from each other.

For example, if your population as number of particles that are large in diameter and the number of particles that are smaller in diameter is correspondingly lower.

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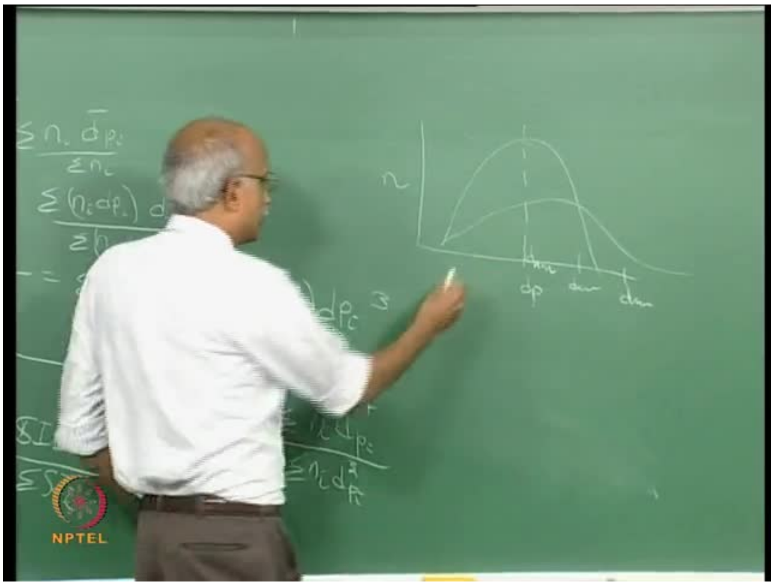

Then the, well, let me sketch this. Suppose that you have a population, this is d P verses n, and the population is such that you have two populations with the same mean diameter, number mean diameter, but in one case, the population is normal, and let say that in the other case, even though the mean diameter is the same, it as a some much wider tail towards the larger sizes. In this case, the number mean diameter would be here d nm with area mean diameter would be where? Could be very far to the right, because these particles even though they are smaller in number, from an area view point, they could be contributing quite a bit.

So, for a population like this, your number mean diameter may be here, your area mean diameter may be here, your volume mean diameter may be here and so on.

And similarly, if you have a population that has an enrichment of finer particles, you may see that again the number mean may be here but you may see that the area mean is over here, the volume mean is over here and so on.

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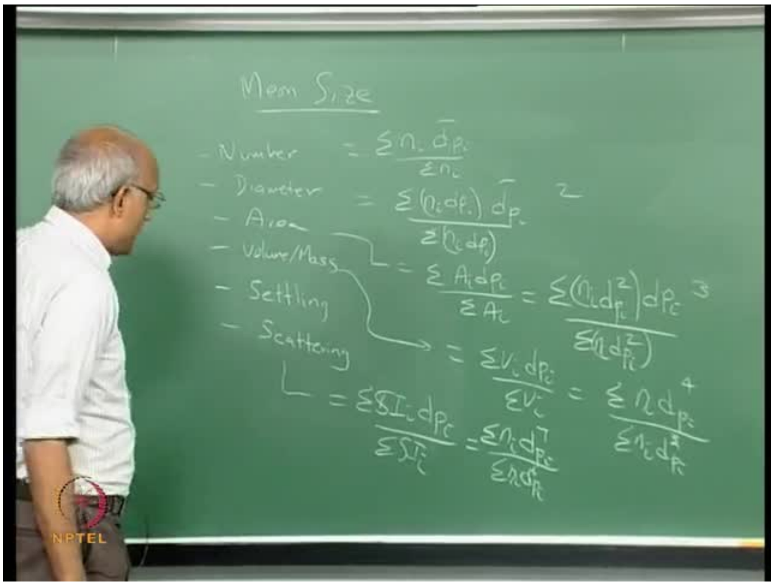

So, the nature of the particle size distribution really affects the relationship between these diameters. Now, if you have, let say perfectly normal distribution, then would you expect is movements to be equal? I think they are likely to be much closer together. There will still be a difference between them, but the more the closer to normality a population is the more similar these diameter values are likely to be, and the further it is, for example, log normal population, what would you expect? In a log normal population, the higher moment diameters are correspondingly finer or smaller than the lower movement diameters.

So, you will definitely see a scaling of these mean sizes of particles based on which moment you take, and by taking the scattering mean diameter, you will estimate essentially the smallest size for that particle population compare to taking a simple number mean.

Now, again the mean is a measure of central tendency. Similarly, you can estimate standard deviations in the data which will again tell you how tightly cluster the population is around the mean diameter verses being more widely dispersed from the center of the population. When you are actually trying to make powders, for the most part, your objective will be to make a distribution as tight as possible.

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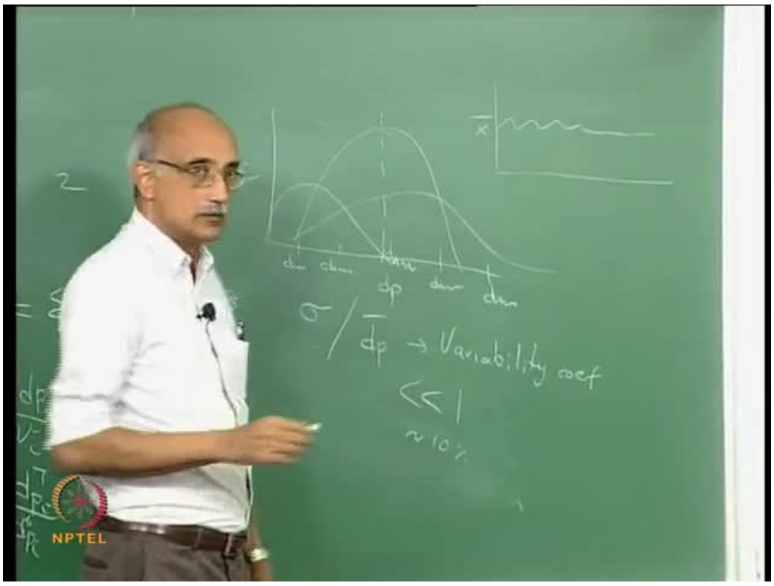

And one of the key parametrics in the context is the ratio of mean diameter d P bar and the ratio of the standard deviations sigma to d P bar is a key variable for you and this is call the variability coefficient. In a well controlled manufacturing process, you want to make this much much smaller than 1. In other words, the standard deviation in the population compare to the mean of the population must be very small. As a thumb rule, you use a 10 percent rule.

If the standard deviation exceeds 10 percent of the mean value, you have a population that is basically out of control or it is a population that is normal, one of the two. If it is a normal population and you are getting sigma by mean values that are greater than 10 percent, then you have to do something to improve your process control and bring it back into a situation where you have a significant reduction in your standard deviation parameter.

The other thing that we do with these values is statistical process control where you actually track these values as a function of time. So, you would take, you would measure your X bar value or d P bar value which is your mean size as a function of time and you will also measure your variability sigma as a function of time, and you will do statistical controls to ensure that they stay within certain limits.

So, this SPC methodology is a very critical aspect of using particle statistics to establish a control over the process. So, this is an important aspect that we will study later one of the lectures in this course under the general title of stastical quality control which involves many aspects of particle characterization. So, will stop at that point, any questions? So, I will see you at the next lecture.