Particle Characterization

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Module No.# 03

Lecture No. # 11

Morphological Characterization:

Acoustic Attenuation Spectroscopy

Welcome to the eleventh lecture in our particle characterization course. In the last lecture, we were discussing particle size distributions, and we noted that, size distributions can be obtained in essentially, a tabulated form. Most particle counters will give you count of particles by size, in various size channels. They will report differential counts of particles only in that channel as well as cumulative counts of all particles, that are larger than a particular size. And, we noted that, these data can be analyzed statistically, to obtain various mean particle sizes; and also that, there are certain particle size distributions that occur in nature, or due to specific manufacturing processes that are employed. And, the three most common distributions are the normal distribution, log normal distribution and the power log distribution.

Again, it is important to realize that, differential counts and cumulative counts are very important in controlling many different processes; but in terms of which one we actually use, for a specific application, it depends on the sensitivities that are involved. For example, when we talk about atmospheric aero salts and their health consequences for people… Now, typically, what we care about are, particles that can be ingested in to our system; our noses are actually excellent filters.

So, they will typically, not allow large, coarse particles to enter; it is the finer particles, it can actually penetrate through our mucus membrane and actually, enter our lungs or, or digestive system and so on. So, in, in this case, the most relevant particle count is, all particles that are smaller than a certain size; because there is usually, a minimum size of particles that can be retained in your nostrils and any particles that are smaller, can penetrate through and enter your system and possibly cause harm. So, an atmospheric scientist would be typically interested in particle counts that are smaller than a specified

size. On the other hand, if you look at a process like microelectronics manufacturing, there is a clear correlation, that has been industry, established in the industry over time, regarding the size of contaminant particles and the probability of causing a defect in the product.

And, here, it turns out that, any precision product or microelectronic product has a threshold size, above which a particle will cause a failure; but, below which the particle is essentially, benign for the product. So, every product, whether it is a wafer or whether it is a hard drive or whether it is an integrated circuit, they all have this characteristic threshold dimensions, below which no harm is caused by the particle; but above which, the, the defect probability quickly rises to 1. So, in such a situation, obviously, a contamination control specialist, someone who is looking after the health of the product, from our particular view point, would be most concerned about all particles that are larger than a certain size.

So, in that case, the cumulative counts become very important. So, the key thing to remember is, a particle counter gives you a lot of data. You have to be careful in selecting, which data are the most relevant and most appropriate for your process. Now, dealing with discrete data is always difficult, because, because of the discontinuity in the data. You know, dealing with tables of numbers is always more difficult than dealing with a function, an equation or a graph, right. So, you always want to convert the set of discrete data in to a continuous function. Now, when you try to represent particle size distributions by a continuous function, rather than tables of data, the error involved in such a procedure, is directly related to the number of particles that you are actually counting in your system. As the number of particles increases, the relative error in assuming a continuous function decreases. For highly dilute systems, it is very risky to interpolate and fit a functional form to a set of data, because the uncertainty in the data, in a particle counter, increases with a reduction in the concentration of particles in the fluid that you are measuring, or, on the surface that you are measuring.

And, so, the representation by a continuous function must be done carefully; but, when it can be done, it does enable you to fit a model to a distribution, which then allows you to do various interesting things, in terms of optimizing the, the particle size distribution. Again, in some cases, you may want to push the particle counts to a lower size; in some

cases, you might want to push it to a larger size, or, in some cases, you just want to establish a very tight control over the mean of the, of the particle size.

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So, when we try to fit a particle count data using a function, now, typically the counts are given as size range or delta in the particle size versus a differential count delta n, right. So, for various values of delta d p, we get values of, let us say, the differential count of particles in that channel. Now, how do you take this data and convert it into a continuous function form? Well, one way to do that, is to define something called d f, which is a

frequency function. It is the fraction of particles in a particular size range. So, you can look at this as, delta n over n, corresponding to any value of the particle diameter d p, where delta n, or, you can even represent this in differential form as d n, corresponding to this differential channel between d p and d p plus d d p divided by the total number of particles n. Now, what you can do is, you can represent this d n as d n by d d p multiplied by d d p.

So, simply dividing and, and multiplying by the same parameter d d p, which is a differential in particle size corresponding to that channel. So, when you introduce this into your equation for d f, this becomes, d n over d d p over n times d d p and this parameter d n over d d p divided by n is what we will simply call f; f of d p times d d p. So, the final equation that we come up with is that, d f which is a fraction of particles in a particular size range is the frequency factor f of d p, which relates to how many particles are present in a particular size range, multiplied by the width of the size range itself.

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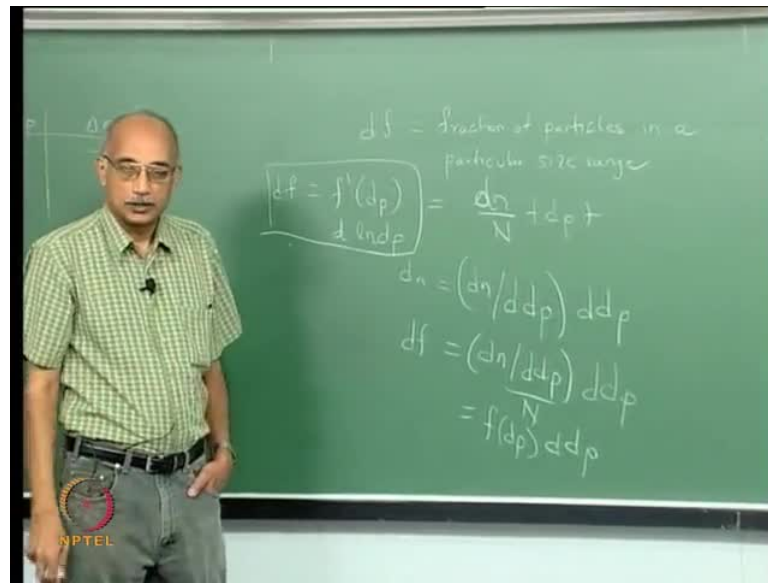
Now, once you have established the value in this form, integral of d f from the lowest measurable value to the highest measurable value must be equal to 1. Now, as we saw yesterday, when we have a log-normal distribution, you are better off using geometric means as well as logarithmic values. So, in that case, the formula in this, in this particular instance would be that, for a log-normal distribution, you would use, d f equals some f prime of d p times d l n of d p. So, you would use this formula essentially, for a normal distribution and you would use this formula for both a log-normal as well as a power log distribution. And, in that case, your integral of d f would still equal 1, whether or not you are dealing with a normal or a log-normal distribution.

Now, suppose you want to know all particles that are, let us say, between sizes a and b. Now, in a, in a differential table, it is very easy to do. You essentially, just take those two numbers and count all particles between them. But, obviously, you can imagine the difficulty that, the sizes here, may not correspond with the interval you are looking for, right. So, depending on the granularity of your channels, you may not get the information you are looking for. For example, if the table is only giving you from, let us say, from 0.1 to 0.2 is one channel, 0.2 to 0.4 is another channel and 0.4 to 1 is another channel.
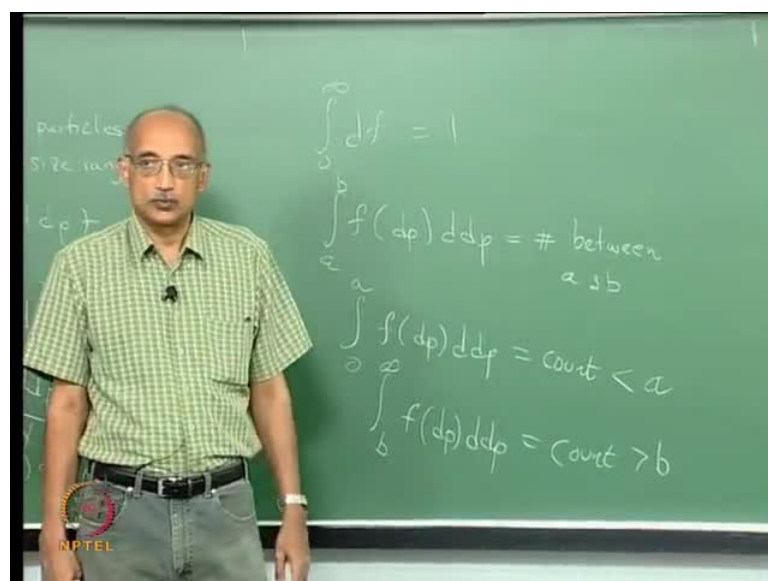
But, you really want to know the number of particles between 0.5 and 0.7, right. Obviously, you cannot use it, do that using the tabular form of the data. That is where continuous functions become important; in order to be able to interpolate, as well as extrapolate the data that you have. So, if you want to obtain that information, once you have formulated a function, it becomes simple. So, integral a to b of f of d p d d p will be the number between a and, sizes a and b. Suppose, you want to know all particles, that are smaller than a size a. All you have to do is, integral 0 to a, f of d p d d p and this would be the count that is smaller than size a. So, this is the kind of analysis that an atmospheric scientist would typically use or a hygienist. Integral b to infinity of f of d p d d p, it gives you the count larger than, of all particles, that are of sizes larger than b. And, this is what we refer to as a cumulative count. And again, in most manufacturing applications, where finer particles typically cause you more problems than, sorry, coarser particles cause you more problems than finer particles, this way of calculating counts is much more applicable and relevant.

So, there is certainly a lot to be gained by fitting a continuous function to particle size distribution. But, always be aware that, the, your confidence level in your fate, will be substantially increased, if we collect more data. So, the more concentrated the suspension is, the better. However, there is an inherent conflict there, because laser scattering techniques are based on essentially, principles of light scattering from a single particle, which means that, the more dilute the sample, the better the accuracy in your measurement, right. So, you have to balance these two requirements.

So, you want to get as concentrated a suspension as possible, in order to improve these statistical accuracy of your analysis. But, you want to keep the concentration lower than the limit at which coincidence is, starts happening. Basically, what would happen is, if we have too many particles that are too close together, instead of single particles being detected by the laser, you may have multiple particles, that are essentially screening each other; and that is called a coincidence error. And, that leads to a significant reduction in the efficiency, with which particle counts that are obtained in a, in a scattering, light scattering type of particle counter. So, we have to balance that very carefully.

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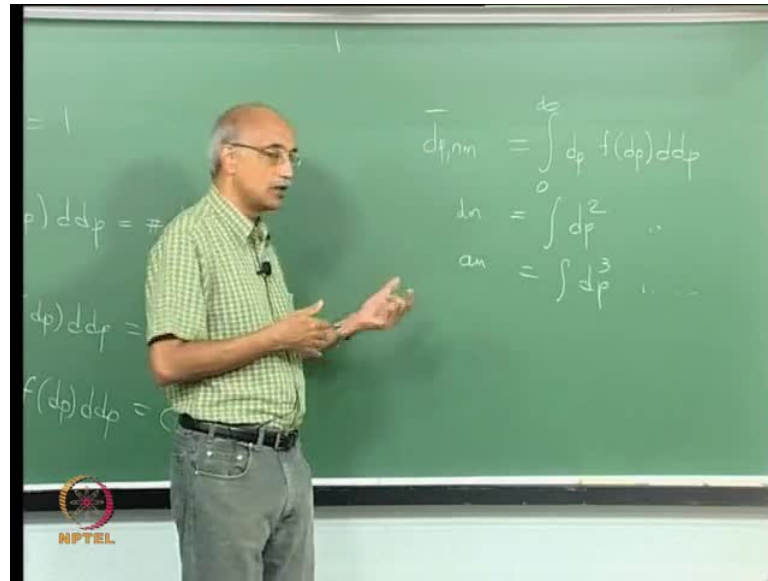
The mean size statistics that we looked at last class, for discrete data, can also be obtained in this, using integral formulations. For example, your d p number mean would be simply equal to integral 0 to infinity of d p, f of d p d d p and your diameter mean would be integral of d p squared; your area mean would be integral of d p cubed and so on. So, these are essentially, the continuous function counterparts to the, the differential analysis of mean size that we discussed in the, in the last class. And, of course, the variations or the standard deviations can also be calculated accordingly, both for the discrete form of data, as well as the continuous representation of particle size distribution. So, the key point in all this is that, particle size measurement in its essence, is a single particle measurement, particularly if you are using a technique like light scattering. But ultimately, the use of particle counters and particle size analyzers is not for looking at a single particle, but rather, an entire population of particles.

So, when we try to extrapolate the technique from usage for a single particle, to an assembly or collection of particles, you have to bear in mind the fact that, there are errors involved. And, when you are trying to obtain a size distribution, as far as possible, try to refer back to some standard spectra that are available; just like you do for, if you have used f t i r spectroscopy, right. You, you obtain a characteristic of the material that you are looking at, but it is very difficult to interpret the spectrum as such. What you do is, you compare it to a reference spectrum in a library and then, you identify that, this material has a signature that is closest to this reference material in my library. So, it must

be that material. Similarly, when you obtain particle size distributions, as far as possible, try to refer back to the standard size distributions, and assign a distribution to a particular population of particles. This will enable you to, also understand the mechanisms of formation of these particles, because as we discussed in the last class, each characteristic size distribution has a specific source, or combination of sources associated with it.

So, the particle size distribution curve will actually provide you a lot of information, not only about the counts and sizes of the particles that are present, but also, the history, of how these particles came to be. So, it is very powerful data, but it must be carefully analyzed and managed. Now, I mentioned that, one of the limitations of light scattering based particle size measurement techniques is the fact that, they require a highly dilute sample. But in practice, there are many suspensions that must be analyzed in a concentrated form. For example, if you are using a lubricant in a machine and you want to control the level of particulates in that lubricant, to prevent wear of the machinery, for example, then, you really do not have the luxury of diluting that material. You want to measure it as is. So, when you talk about measuring suspended particles in a lubricant, that is used in a, in a machine tool, it is a highly concentrated suspension.

Another instance of that would be, again, in semiconductor manufacturing, there is a process called C M P, chemical mechanical planarization, where a slurry of particles is used to polish a surface to very high surface finishes. Here again, in a slurry, you are forced to use high concentrations of particles. They just, just would not work, if you are using a low concentration. But, if you take the slurry and try to dilute it, in order to measure its particle size distribution, you actually change the properties of the slurry itself; in particular, parameters like a zeta potential could be affected, which can cause differences in the size distribution. So, by diluting the sample, you actually change the size distribution characteristics of the sample. So, this is another example, where you do not really want to dilute the sample. But, you still want to measure size distributions over a very wide range, ranging from nanometer to millimeters. Another example would be emulsions; oil in water emulsions, various other types of emulsions are commonly used in many different applications. Now, when you look at emulsions, again, they tend to be fairly concentrated in nature and they tend to have optical transmission characteristics, that are very close to each other.

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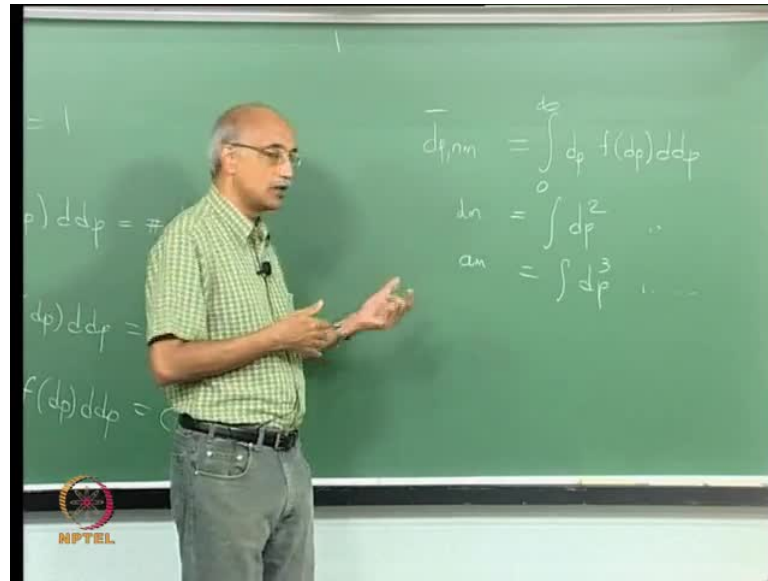
In other words, you know, we have a particle in a fluid; the optical characteristics of the particle are very distinct from the optical properties of the fluid. So, you can easily distinguish them. But, when you have oil suspended in a, in water, let us say, the optical characteristics are not so distinctly different, that you can clearly say, just based on light scattering, whether this is oil or whether this is water; especially as the droplet size becomes smaller and smaller. And, so, in such cases, you really cannot use light scattering as a technique to measure particle size distribution. So, what is our alternative? One of the techniques that is in widespread use, is the use of acoustic waves, to accomplish the same purpose. So, instead of irradiating the fluid with a light source, essentially, you transmit an acoustic field to the fluid.

What happens? Well, you have to imagine this as essentially, three phases that are being present, now. You have the fluid phase, you have the particle phase and then, you have the sound wave, that is propagating through this suspension. The particles that are present in the suspension, act as discontinuities, which essentially interact with the sound wave, that is progressing through the fluid. And, they cause the sound waves to be scattered. Some of the sound energy is absorbed, but basically, there is an extinction, or attenuation of the acoustic field, due to the particles that are present in the suspension. Now, and this is basically called acoustic attenuation; that is a name for the phenomenon that is going on. Now, what are the effects of this acoustic attenuation? A - some of the sound energy will get absorbed by the particular phase that is present. And, as that

happens, there will also be a heating up of the particles. So, a temperature gradient will develop, between the particle and the fluid. And, some of the sound energy will also be absorbed by the fluid medium itself; and again, that can cause, more of a macroscopic heating effect.

So, lot of the acoustic energy gets dissipated through absorption, either by the particles that are present, or by the fluid itself; but, some of the energy also gets scattered in various directions. So, if you look at the transmission characteristics of an acoustic field, it should give you a pretty good idea of the particle concentration in the suspension, as well as the size distribution of particles in the suspension; because, just like with light scattering, acoustic wave scattering is also very much affected by the size of the particles, the shape of the particle, the concentration of the particle and so on. So, acoustic scattering is a technique, which is superior to light scattering for concentrated suspensions; because unlike light scattering, which essentially requires a single particle interaction with the light source, in acoustic scattering, you can actually transmit the sound wave through a, a concentrated slurry of particles and you can still de-convolute the phenomena that are going on sufficiently, so that, you can still establish a particle size distribution. So, this technique of acoustic attenuation is predominantly used for concentrated suspensions. But, there are some uncertainties in the data.

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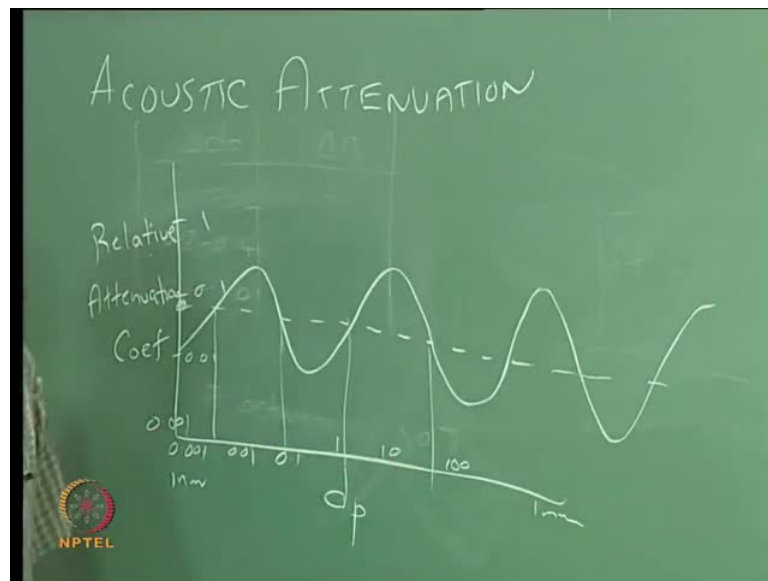
If you look at the effect of particles on attenuating acoustic light, you see, you see a very interesting trend. If you plot particle size on the x axis and a relative attenuation coefficient on the y axis, what sort of trend would you expect? If you recollect, when we talked about light scattering, for example, as the particle size increased, the scattering intensity increased as d p to the power 6. So, the attenuation also increases as d p to the power 6, in the case of light scattering. In the case of scattering of an acoustic wave, when we look at ranges of size here from, let us say, 0.001 microns to 0.01, 0.1, 1, 10, 100, upto 1 millimeter, right. So, this is from 1 nanometer to a millimeter, and let us say that, these are relative values and again, they can range from 0.001 to 0.01, 0.1, 1 and so on.

The trend that you see, is interestingly, now, cyclical in nature. Now, that is fine. I mean, it just happens to be the way, that acoustic waves interact with the particles in a suspension; but the problem now is, what you are measuring, is this attenuation coefficient, right. So, when you look at that, how many possible values of d p can you predict for each spectrum? So, you just draw a horizontal line and you can see that, it is multiple; because if, if you are just measuring this, then, this could be the size, or this could be the size, or this could be the size, this could be the size, right.

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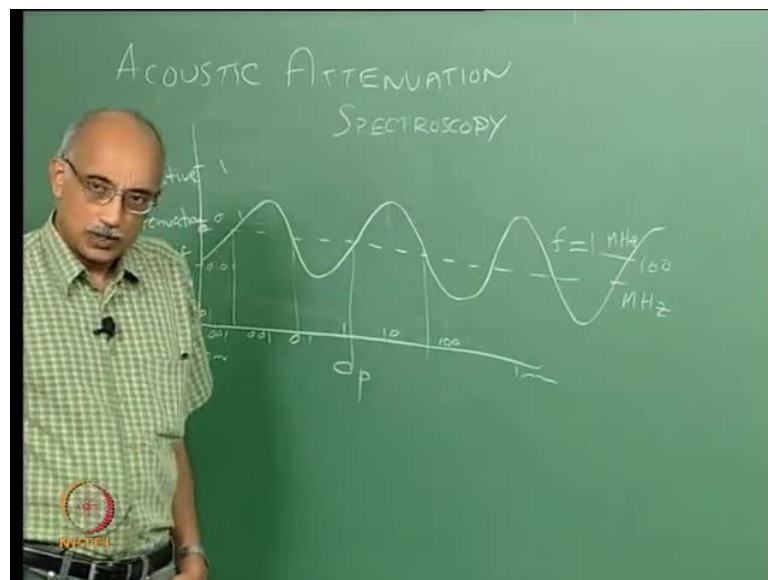
So, there is a lot of uncertainty. So, based on a single acoustic attenuation measurement, you cannot conclusively say, what the size of the particle is. Now, if you have some prior

indication that, this particle is in the nanometer size range, then, yes, we, in a single measurement, and, just look for a, a particular value, that is in the size range you are expecting and you can conclude that, that is the actual size. But, if you do not know anything about the particle, powder that you are looking at, then, there are multiple values. Now, this is for, let us say, a frequency, ultrasonic frequency of 1 mega Hertz. So, how do you resolve this? How do you deal with this? And, that is where, this technique called Acoustic Attenuation Spectroscopy comes in. The idea is clever, basically, say, what, what we do is, instead of only transmitting the acoustic wave at a single frequency, we actually transmit a spectrum of frequencies, all the way from, typically from, 1 mega Hertz up to a 160 mega Hertz.

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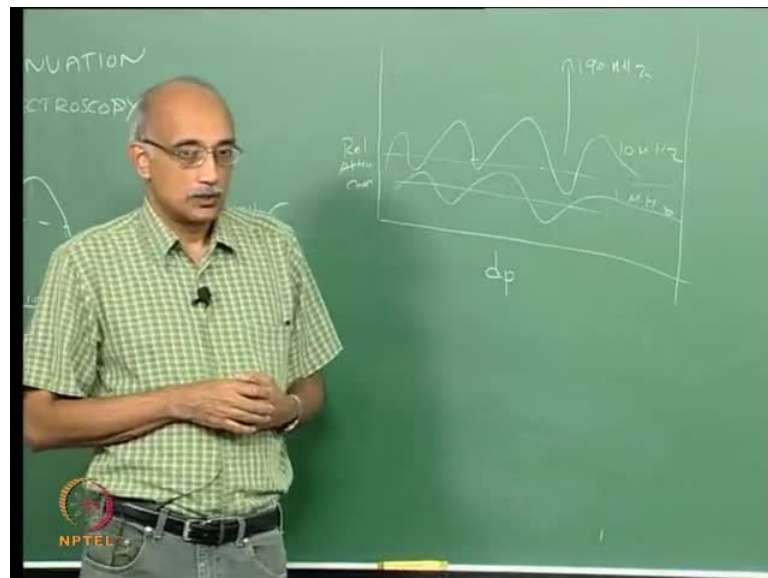
Now, what will happen is, at each of these frequencies, you will see a different spectrum. So, this may be the spectrum corresponding to 1 mega Hertz. At a higher frequency, you will see a greater attenuation. So, this may be at 10 mega Hertz. And so, you keep going like this, for as many frequencies as your system can handle. You can essentially, have an infinity of frequencies, that you transmit and measure at.

Now, what happens? Again, if you, if you use the same methodology when you are plotting d p versus the relative attenuation coefficient, based on one frequency, you may have detected, possibly four different sizes. If you test at another frequency, then, in principle…By the way, these peaks move to the left. I have not sketched it very well;

but, as you increase the frequency, thus the, the peaks that you see, slowly move to the left. In other words, there is a relationship between the wavelength of the light and the diameter of the particle that decides how attenuation happens. So, because of that, in principle, if you just test at two different frequencies and look for the one common diameter, that is detected at the two frequencies. In other words, is that an, when you, when you again, you look at the possible sizes corresponding to the second frequency, in principle, there should be only one size, at which there is convergence between these two.

That is, assuming, absolutely no measurement errors and no possible intrusive effects that can affect your sound transmission and so on. In practice, it is not possible. And that is where the spectroscopic technique comes in to play. As you keep increasing the number of frequencies that you test at, and you keep measuring possible sizes, you know that, you obtain based at, at this various frequencies, eventually, you will identify that one unique size, that is present in all these spectra. And that, you decide, is the actual size of the sample, of the particles in your sample. Now, the limitation of what I just talked about is, this only works for mono-dispersed samples.

In other words, there is only one size. Now, what happens, if there are multiple sizes in the population? You know, when you have a particle size distribution, can we still use this technique, such each size particle will attenuate the acoustic signal differently? So, the problem becomes multi-dimensional. However, there are mathematical inversion techniques that you can use, to extract even the size distribution of a poly-dispersed population; but, the procedure is a little more complex, when you are dealing with multiple particle sizes.

The first thing is, based on the, the physical mechanisms that are happening, you have to predict an attenuation spectrum. In other words, by using conservation equations of mass, momentum, heat, by using constitutive relations such as the equation of state, and by looking at the fluid mechanical properties, constitutive relationship, such as strength, stress versus strain relationship and by using the wave equation that describes how a wave interacts with media, you can actually predict, for a hypothetical particle size distribution, you can predict, how these attenuation spectra are going to look like. So, that is a step number one. The step number two is, you actually make the measurements. You know, the same, these measurements. You irradiate your sample with, the acoustic

frequencies over a wide range, obtain the spectrum and then, you compare the two and look for the difference or the delta.

If the difference, if they do not match exactly, you tweak your model. So, you take your experimental data as being absolute and you try to tweak your assumptions about the particle size distribution, until you get an exact match. It may sound complicated, but mathematically, it is not that difficult. There are algorithms, which will essentially, optimize the fit, to get minimum deviation between the predicted particle size distribution and the measured particle size distribution. And, that is the way it is done, in practice. So, companies that make particle size analyzers, based on acoustic attenuation spectroscopy, for example, Malvin instruments, has, has such an instrument. The equipment comes with a pre-built-in algorithm. So, all you have to do, is specify the fluid and specify the particle and specify the frequency range that you want to employ, and the size analyzer will immediately generate a prediction of the attenuation spectrum, for a hypothetical particle size distribution.

So, it is all done for you. You do not, I mean, that, that the software already does the theoretical prediction and then, the instrument will do the actual size measurement by transmitting an acoustic field. It will then do the comparison; it will do the iteration. The whole thing will take about ten minutes. And, you will get an, a particle size distribution using an acoustic attenuation spectrum device. Again, the power of this technique is a very wide range, because this is very useful to us, right. There is no, there is no attenuation of the intensity by particle size. You can go from very, very small sizes to very, very large sizes, without there being a significant systematic difference in the attenuation co-efficient, unlike in the light scattering case, where there is clearly no, no way you can use light scattering for particles that are smaller than a certain size. Inherently, there are no limitations to using this acoustic technique, over a very, very wide particle size range.

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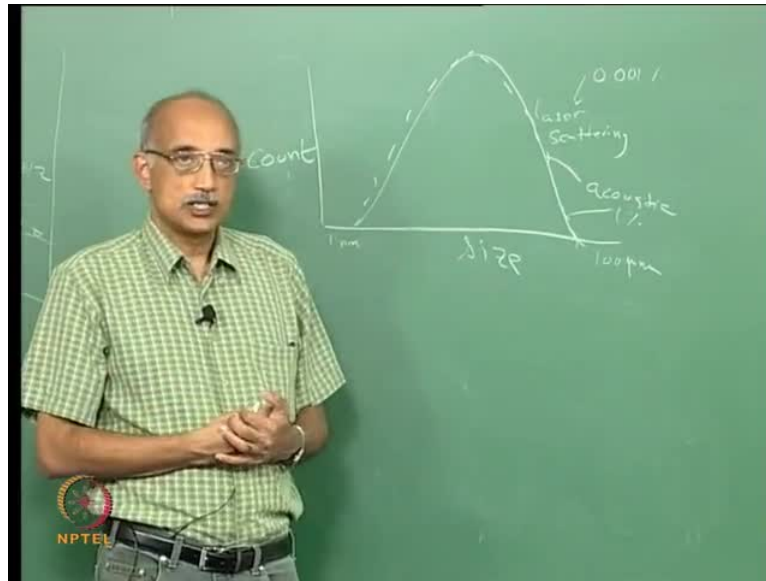
Now, the limitation in this case, is that, it does not work well, if you dilute the sample too much. So, it is exactly the kind of, the mirror image of light scattering based technique, where the techniques do not work well, if the concentration gets very dilute. So, in fact, correlation measurements have been done between the two types of instruments. So, if you take the same suspension and you plot size versus count, and let us say that, you are looking at a range, from again, 1 nanometer upto, let us say, 100 micrometer. So, let us say, you get some spectrum, based on laser scattering. You take the same solution, and you subject it to the acoustic method of monitoring, will you get the same data? Yes, you will. So, let us say that, the acoustic device gives you this data.

This is the acoustic attenuation spectrum based methodology. But, what is the difference? The difference is, with laser scattering, you would have probably measured at a dilution of 1 to 1000, roughly. So, the volume percent of particles in this may have been, let us say, 0.001 percent, in order to get good data. But, the sample that is used for the acoustic technique, the concentration can be as much as 1 percent. In fact, it has to be as much as 1 percent, in order for the data to match. If you take this solution and you start diluting it, then, you will see that, the distribution you are getting in the acoustic device, will start deviating from the distribution that you got in the light scattering device. Similarly, if you take the solution that you used for the light scattering measurement and you start concentrating it, you will see that, it is start deviating from the measurement used in the acoustic attenuation device. So, you will never get exact

coincidence, at the same concentration. But, you can get, virtually, the same data from the two instruments, if, in one case, you dilute and in the other case, you concentrate, the sample. And, in fact, the way that you want to use an acoustic particle counter, is by determining the concentration, at which you start getting stable values.

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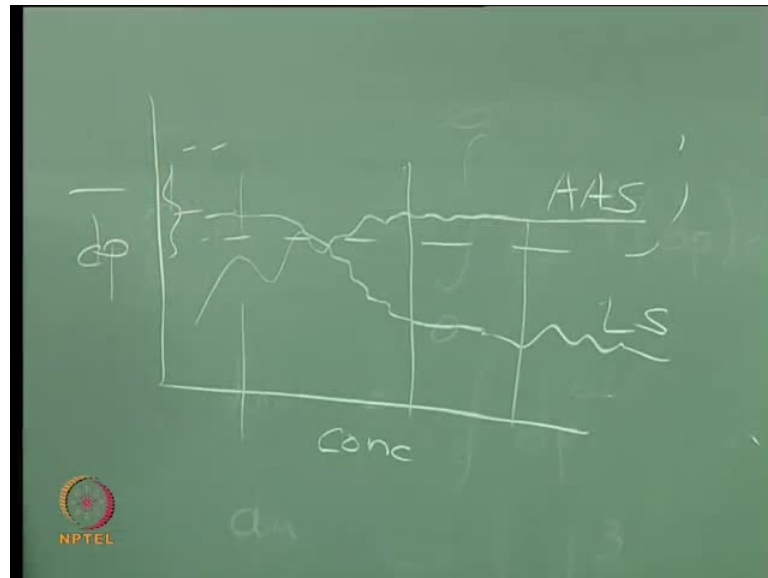
So, for example, what you should plot is, something like, concentration of particles versus the mean size. What you will find is that, upto a certain concentration, there will be deviations in the data; but eventually, it will stabilize. So, this is a characteristic behavior of an acoustic attenuation spectrometer, for measuring particle sizes. If you repeated this, in the case of a light scattering device, the trend you will see will be, the exact inverse. So, what you will find is that, you get a behavior, which shows significant deviation at the higher sizes, but, gives you a very stable data at lower concentrations. So, the stable size that is predicted by both methods, will be very close. But, if you are not careful, you may have measured at the wrong concentration and got completely different values.

Depending on the concentration, if you use the same concentration, then, it is very unlikely that, you will get the same data. In fact, there will be, for the most part, a significant deviation. Coincidentally, there may be a point, where they coincide, but, even that would not be the correct value; because this value is obviously, not the right value. The asymptotic value that you see, is the actual mean size. And that is why, it is, it

is very important to understand the principles involved in various measurement technologies and make sure that, use the one, that is appropriate for our sample.

I mentioned that, the acoustic size analyzer could be used for emulsions, and in fact, some very careful, quantitative, correlation work has been performed using s e m t e m image analysis and particle size measurement, using acoustic attenuation. And, you can see a virtual, one to one correspondence between the particle size distributions that are predicted by the two methods. So, it is a very powerful method. The usage of an acoustic attenuation spectrometer is not as widespread as light scattering, primarily because, in most conventional applications, you are dealing with dilute samples. But certainly, in any situation, where you have to deal with the concentrated sample, the acoustic based techniques will work much better for you.

Ok, a couple of other points, such as, we should look in to, before we complete our discussion of particle size analysis. When we measure size of particles, in a suspended in, particularly in air, there are really three characteristics sizes that you can specify. One is, what we call a primary sphere diameter. The second is agglomerate diameter and the third is crystal size. Now, this distinction becomes especially important, as you start approaching the nano sizes. Nano particles have a clear tendency to agglomerate. So, when you are trying to measure the size of a nano particle, it is very, very difficult to do so; because, by the time you get ready to do the size measurement, it has already moved, found its nearest neighbor, and attached itself to its nearest neighbor. So, every time you try to actually measure the size distribution of a sample containing nano dimensional particles, what we find is, what we are really measuring, is not the size of the individual nano particle, but rather, the size of a conglomerate of such particles that are adhered together.

And, even in the case of a single nano particle, the crystalline structure that is present within that particle, is different in size from the particle as a whole. As we will see later on, the structure of a particle, really encompasses three different regions - the crystal or the core is, is the interior, but, there is also, what is known as a subsurface region and a surface region, which act as an interface between the crystalline structure of the particle and its surroundings. And so, these three sizes are important to characterize, in, when we are trying to determine the shape, well, in particular, the size of nano particles. So, in the next lecture, we will start out by discussing these aspects in a, in a little more detail,

because, it is very important to understand, if you want to extend our particle characterization technologies to the nano dimensions. In the next class, we will also work out a few problems, that illustrate some of the principles that we have talked about so far, in our lectures.

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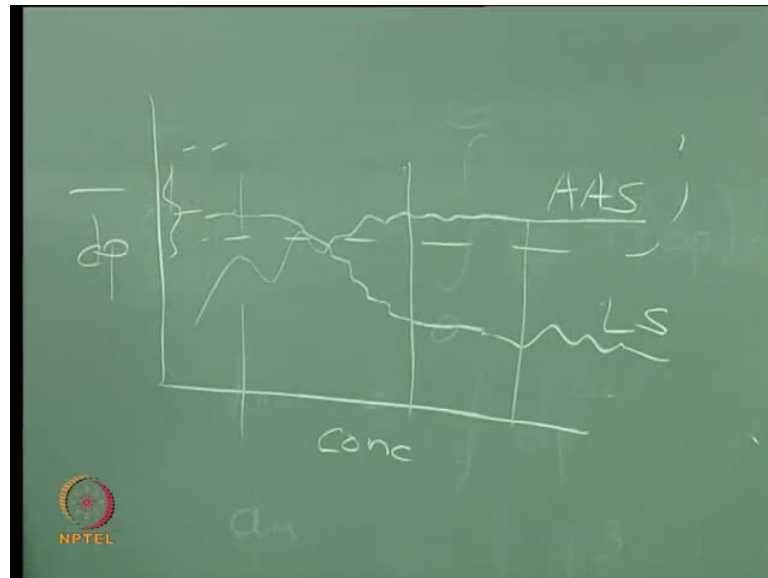
There was a question before the class, from one of the students, about text books to be used for this course. Now, particle characterization is, is a very widely studied and researched area. So, I would not recommend a single text book to cover everything related to particle characterization, but, a good book to begin with, is the one by J K Beddow. It is called Particle Characterization. It was written back in the 60s. I think it was reprinted once, in the 80s. Beddow is considered like the godfather of particle science and many of the things that we have talked about in this course so far, particularly related to shape analysis and some of the size analysis methods, that are based on direct inspection of particles were actually laid down by Beddow and his fellow researcher.

So, it is a very good text book to read, in order to understand the history of particle characterization. But, some of the methods that we have talked about more recently, based on laser light scattering or acoustic scattering, are really not covered in that textbook. What I will do is, in, in one of the later lectures, I will provide you a list of references that will cover the various sources that I have taken my lecture material from.

I also encourage you to read current literature; I mean, there are lot of publications in journals and conferences, in the areas that we have discussed, and that we will be discussing in future, particularly Nano-technology. It is, it is an emerging area and people are constantly discovering, as they go along. So, the problem in Nano-technology is, by the time you publish a text book, it is already obsolete, because, that, the technology is moving so fast. So, really, the only way to keep on top of Nano-technology is to read current papers and I certainly encourage you to, to do that, wherever, whenever possible. Let us stop at that point. Any questions? Right, I will see you at the next lecture, then.