Environmental Geomechanics
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Lecture No. 55
Pore-structure characterization - I

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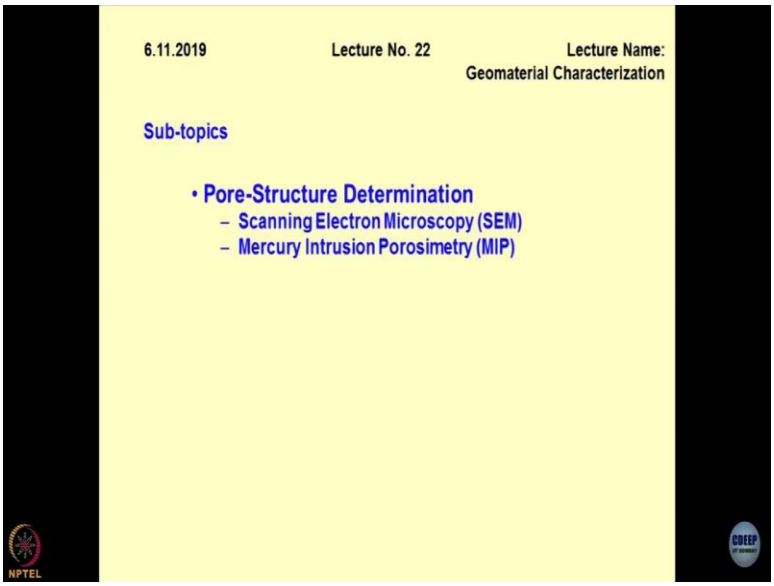

Here, I will be discussing the pore structure determination; you must realize that most of the mechanisms that occur in geomaterial largely depend upon its pore structure or in other words, pore size distribution. Without knowing the pore structure of geomaterials, nothing can be done much. And this is a very intricate work or subject I would say. So, normally we use two types of techniques. One is the scanning electron microscopy, which is not a very quantitative way of finding out the pore structure of geomaterials.

Incidentally, the MIP is done. MIP is the mercury intrusion porosimetry. Well, there is a school of thought which keeps on evaluating these techniques vis a vis each other. But the point is that there is nothing better than these techniques which is known to human beings at this stage and which are affordable and feasible. So, I like to give you some feet interact some ideas about what the SEM, MIP is all about.

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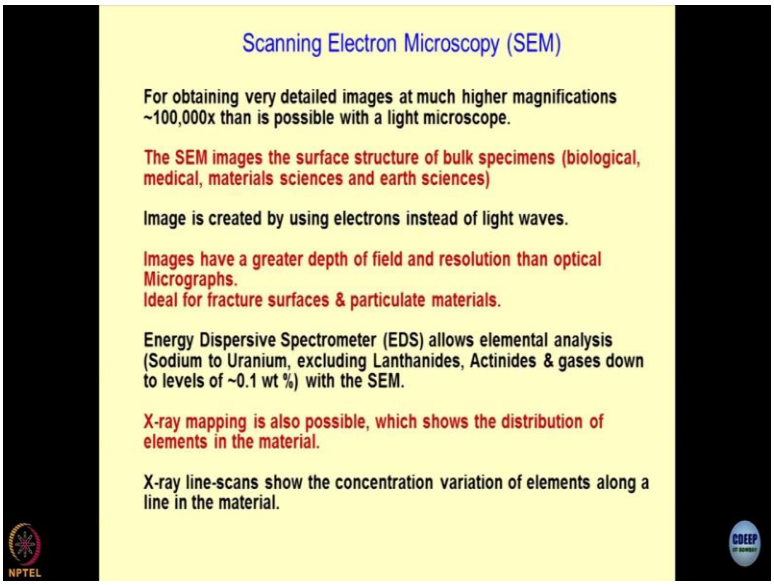

Some of you would get a chance to use these techniques in your research career, and some of you who do not get a chance to use them right now might get maybe after some time, or you can appreciate by seeing the YouTube videos and so on. So, it is scanning electron microscopy, what is known as SEM is normally obtained is normally done to obtain the images of the sample at very very high magnifications.

So we can go up to one lakh times of the magnification with the small microscopes curtesy to the recent day electronics basically this technique is used to study the surface structure of the bulk materials and surface features could be the texture of the soil mass, the pores, the cavities which are present, the orientation of the grains of the soils. So, I will show you today how easily the dispersed and flocculate structure in the fine-grained materials can be looked upon by using SEM technique and then how to quantify them.

At the same time, the biological processes which are occurring in the geomaterials, I think I have shown you some of the pictures when you are discussing the biological processes. Biological characterization of geomaterials, where people are interested in seeing what type of EPS is getting formed what type of bacteria is present in the system. What type of deformations is happening in the geomaterials? SEM is used in the field of medical engineering, material sciences, earth sciences, quite a lot. The image is created by the beam of electrons.

There is a simple thing. And one good thing is that we can cover a greater depth of the field and resolution than optical microscopes. And you can study the fractures and the cleavages which are appearing on the grains of the soils. There is a new version of SEM, which is known as EDS, and we call this as energy dispersive spectrometer, and this also gives you the element analysis of the material. So, you can find out what type of elements which are present in the system.

And then by using simple mathematical calculations, you can obtain their percentages also in the oxide form, we can also see how the distribution of the elements has is occurring on the material particularly when you talk about the mineralogical composition. Another question would suppose if I consider a grain of soil, let it be quartz or let it be montmorillonite or kaolin. I would like to see how the mineralogical composition of the grains is changing on its surface.

So, Dr Srinivas Kadali was my PhD scholar who has done very interesting studies by using EDS, and another is Dr Bhagawanji Jha, who has done synthesis of zeolites, and then he wanted to understand, what is the mineralogical composition, elemental composition of the zeolites on the surface. These are all microanalysis, which has been done, and these students have been pioneered in this area. And by using the methodology in which they have developed.

We have done X-ray mapping of the elements which are present in the material and show a process which happens, particularly the alteration of minerals over a period of time. When the minerals come in contact with contaminants. This could be acidic or basic or biological. Incidentally, biological features have been studied by Dr Shashank.

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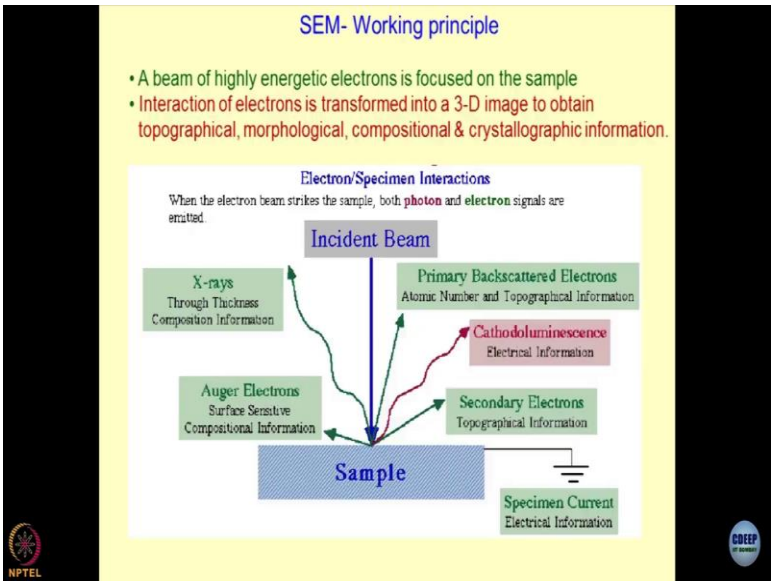

This is the working principle of SEM, a beam of high electric, high energy electric electrons is focused on the sample, and then the interaction of electrons is transformed into a three-dimensional image to obtain the topography, morphology and composition and crystallography of the grains. So, topography, morphology and compositional characteristics I have discussed and crystallography is basically how the crystal structure is changing.

If you Google it, you will realize that the working principle of SEM is quite simple. You take the sample and then bombard it with the electron beams. Whatever the beams scatter out of this if I take out the X rays beam, other electrons, primary, backscattered electrons, secondary electrons, then I can analyze these beams, and I can filter them to obtain the information.

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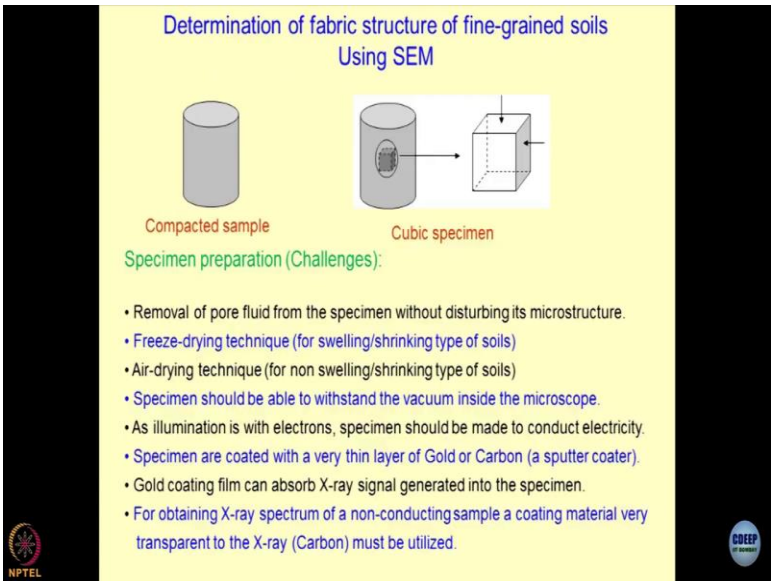

So, using this concept, we will have been doing the fabric structure of fine-grained soils, what we have done is we have compacted the soil sample. Let it be a triaxial sample for that matter, and after the sample has been tested for its shear strength properties, you can take out some small element from the within the sample is about $1cm^3$ cube, and then this $1cm^3$ specimen is looked at from the sides perpendicular sides to see the material heterogeneity in the perpendicular and parallel to the compaction plane.

When you are making this sample by compaction, you will realize that you compact the sample in the vertical direction. So, I would like to see how the grain structure is changing along with the compaction, and this is the lateral direction. So, we would like to see the features of the sample perpendicular to the plane of compaction and parallel to the plane of compaction. And incidentally, the ratio of the two is the form factor which we talked about. So, it appears to be very simple, but truly speaking it is a very complicated way of doing the studies because I am sure when you go to the lab, you take out a $1cm^3$ cubic specimen of clays from the triaxial samples and then make it a good specimen, which can be utilized to study the micro features. It is not a very simple task. And why it is so difficult, I have listed some of it over here. So, when you remove the pore fluids from the specimen, the structure gets changed, because ultimately, the fluid is back between the grains.

So, when you are taking out the sample, the chances are that you are disturbing the whole synergy of this specimen that the surrounding sample. So, if microstructure gets changed, it might be by holding $1cm^3$ cube, specimen by hand or by forceps because you are applying some pressure on this. So, the microstructure gets changed it is not an easy task, as I said the second Is that most of these SEMs they work on the frozen samples or the sample we do not have water.

So, this is where we use the freeze-drying technique. This becomes very difficult when the minerals are swelling and shrinking type. Because the moment you take it out from the sample and keep it in the atmosphere, the evaporation takes place and the sample cracks. So, once the sample cracks, you are not really going to get the real picture of what the sample used to be when it was in the triaxial sample, air-drying technique cannot be utilized here.

So, one has to be very careful. And there are different types of setups which are available to do vacuum drying or the freeze-drying of this material. You freeze the sample. Now the question is when you are applying the vacuum, then the sample should be able to withstand the pressure which is coming from the vacuum also. So, from all sides, there are issues. Another thing is that soils are mostly nonconducting materials for electrons.

And what we are doing is we want to study how the charge gets spread on the sample. That means, I need a surface of the material which is conducting. So, this is another problem. So, what is done is that we normally apply a thin layer of gold and or carbon; this is what is known as a sputtering material. So, to make the sample conductor of the beam. So, this is more of an art rather than engineering making a specimen itself and people have to spend a lot of time.

If your materials are nonconducting, then also there is a problem, and in short, the making of the sample requires a lot of good hands and training. But once you have made the samples and sample the samples which are useful for studying the microstructure.

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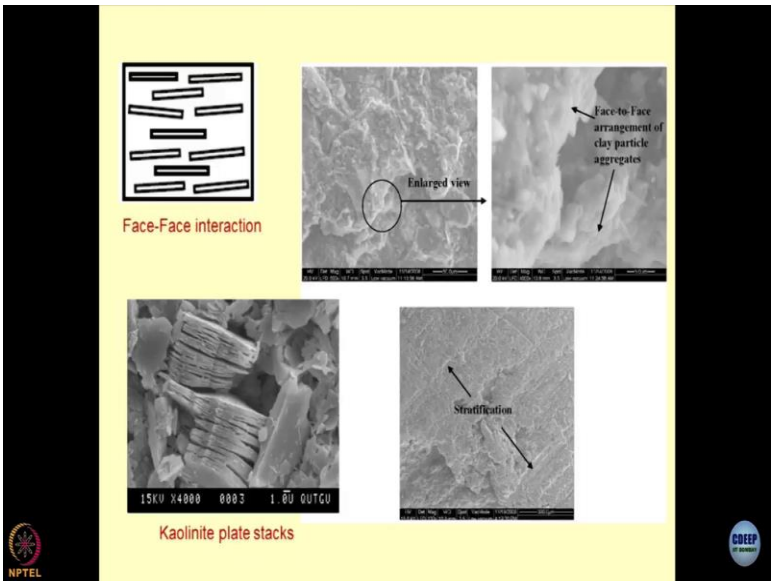

These type of information can be deciphered very easily. So, all of you must have studied how the face to face interaction looks like, but I am sure not many of you have seen it ever. So, this is how it looks like in the SEM images. Now, I hope you can see that this is one clay platelet and these are the other clay platelets which are sitting one over the other. Look at this picture, can you differentiate between the layers of the clay platelets one over the other.

Now, you cannot make out much. You can. So, these type of investigations are to be done to realize how much is the dispersed state of the material is and then one of my PhD scholars Dr Suchith Gumasthe; he quantified the degree of dispersity and degree of flocculation of the material by using impedance spectroscopy and SEM. His thesis was based on this, a very interesting work he has done, and we have simulated how sedimentation occurs in nature in the lakes and the oceans and reservoirs.

Now, this is how it looks like. I hope now you can realize the beauty of the kaolin plates the way they stacked. Later on, we have utilized these SEM images for deciphering a lot of information, I hope you can realize the type of cavities which you see over here are very interesting parking places for any fluid, this could be fertilizer, this could be enzymes, this could be bio enzymes, this could be any type of medicine, pesticide, whatever.

So, I think this is a whole lot of playing with the minerals and making them more worthful. So, the more and more you zoom into the system, you realize the beauty and how much nature is intricate. So one of my PhD scholars Dr Sasmita Sharma, she utilized SEM to realize what type of sediments existing in the sewage and wastewater treatment plants because those type of sentiments have been ignored until now, but for us, the sediments which are occurring in these sewage and water treatment plants are also sediments.

And the whole idea was that if I decant these ponds what I'll be doing with these materials, so, to our surprise, we realized a lot of peculiar formations which occur in these sediments, including the pathogenic as well as microbial and bacterial activity. So, this thesis of Dr Sharma was dealing with as I have said earlier also this was dealing with the social economically generated settlement SEGS, we have termed this. So, a lot of information can be discovered from this simple analysis.

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Now, I am sure that will be intrigued to see how the face to edge and edge to edge interaction of the clay particles appears to be. Can you make out from this figure that how edge to edge platelets are sittings with each other, this is the edge and some another I just coming and sitting over here? So, this is a peculiar system of edge to edge contact. If you see here, this is a face edge combination. So, this is how the microstructure analysis is being done by using SEM.

Now, many times people ask a question whether SEM can be utilized to obtain in the porosity or not the answer is not really because SEM, as you are seeing, is a qualitative technique. This is a pictorial way of demonstrating what exists in the system though there are cavities or the voids which can be quantified, but it will not be very easy to look into the third dimension that is perpendicular to the picture. So, what people do is that they do tomography of the pores, and tomography of the pores is known as for porosimetry.

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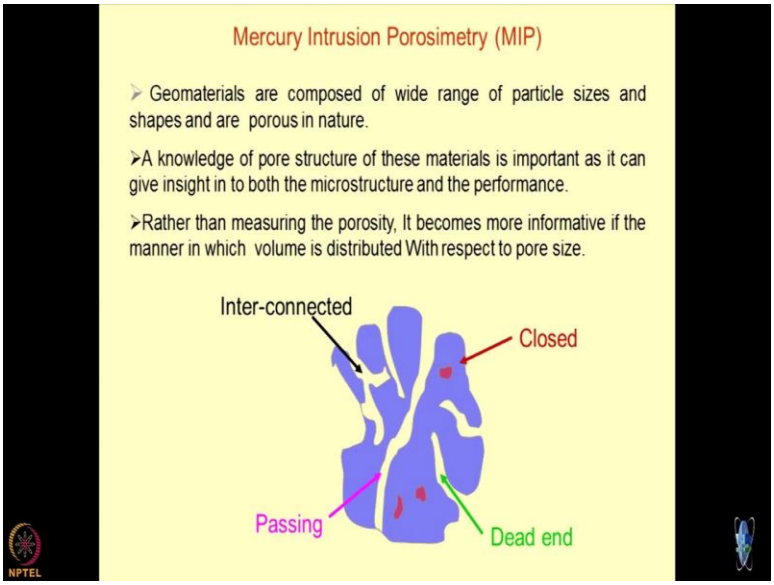

So, porosimetry is becoming very, important in the realm of environmental geomechanics because of the obvious reasons that pores are the ones which play an important role in any mechanism which occurs in the geomaterials. Transport of mass flux alike. So, this flux could be thermal, electrical, chemical, biological, radiological, magnetic, and so on. So, until now, we have been talking about only the particle size distribution in conventional geomechanics.

But truly speaking particle size distribution does not give much idea about the geomaterial characteristics and unless you really talk about the pore structure and the shapes. So, there are a lot of people who are doing fundamental research in modelling the pores why because we can decipher the microstructure of the soil and when I say micro structure basically this is the grain size and their lattice or the fabric we call it how the grains are located in the matrix of the soil. Now, this is a simple model, if you consider a grain of the soil. Now, what you will realize is that there are different types of pores which are present in the grains, there could be a sort of a pore

which is sitting on the surface like a crater, and this is what is known as the closed pore there is nothing which is going to migrate through, and through this pore, it is a sort of a crater on the surface of the grain. But yes, I think when we discussed the adsorption and absorption phenomena, I think this is where I had talked about, the first thing to happen is that the contaminant has to come in contact with the geomaterial which is a physical process. Now, once this contaminant stays over here, the chemisorption will start this will penetrate into the matrix of the grains. Now, in this case, we have dead-end pores, the pore is well defined, but then ultimately, the fluid flow across the mineral cannot occur.

So, this becomes a dead end. We have an interacted pores; these pores are interconnected forming a sort of a network within the particle itself. Then there could be through and through passing of the pores, look at this there is a pore which passing through the grain itself. So, these type of pore arrangement and the ones which I might not have shown over here or the combination of these type of pores exist in the porous materials.

So, this becomes a slightly complicated situation where you would like to find out what the pore structure is what the pore size distribution is, and once these two things, you would like to understand what the porosity of the system and this porosity is going to be the absolute porosity, which cannot be obtained by soaking the geomaterials in water which normally is done, they take the rock samples and the soil sample they soak it in water for 72 hours and then find out the weight,, and then they say that this is equivalent to the porosity.

But the simple logic is as we have discussed earlier also water molecules cannot enter into the finest of the pores which are present in the grains and hence the porosity which you obtain by soaking of material in water is not going to be the true porosity. So, with this premise and the background people have gone into understanding, and they have developed a lot of techniques for doing porous structure modelling or what is known as the porosimetry.

So, when we talk about the porosimetry, the MIP becomes important or this is the most important techniques technique, which is used for determining the pore structure and the name is mercury intrusion porosimetry. And the beauty of mercury is that surface tension is very high or

very small. It is a wetting fluid or non-wetting fluid?. Very good. So, it is non-wetting fluid. So, surface tension is going to be extremely high. That means the smallest ball or the drop of the mercury can still remain in the spherical form.

And the beauty is the more and more pressure you apply the drops of the mercury will start becoming finer and finer, better than water, water is incompressible so is mercury also, but surface tension and the wetting properties of the water make it not fit for doing porosimetry. Now, the question is if I really want to know the finest of finer pores in the material what I should be doing.

I can use gases nitrogen, helium gas which we had talked about when we were doing helium gas pycnometer. So, you can use helium gas to pass through the sample and capture the total porous structure, because the size of the helium gas, nitrogen gas is going to be much smaller as compared to the mercury. So, these are the facts which are normally kept in mind.

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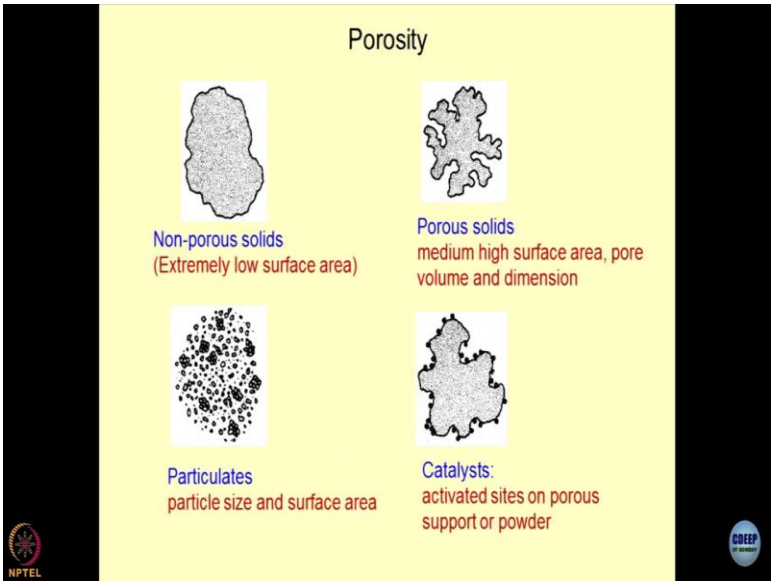

And another interesting thing is, I hope you should realize that porosity is something which could be in any of these forms. This is a non-porous solid for that matter a perfect quartz grain of the sand. Now, this has an extremely low surface area because there is no cation exchange capacity, it is quartz is a very dull material, it has no affinity towards the external environment All right. So, truly speaking non-porous solids are made up of quartz. Quarzitic material.

They have an extremely low surface area, this the porous solid and why porosity because of the interstices which have got created under the grain itself and they have the slightly high surface area, and they have some amount of pore volume and their dimension. Now, this is a particulate system, the dispersed particles in the matrix of the soil mass. So, when you have a particulate system like this, their particle size and surface area vary and we are more interested in seeing what type of particle size and the surface area, the system will give.

Incidentally, when we do porosimetry, we have intentions. One is to have the pore size and second is to obtain the surface area also, ultimately, when you are intruding something into the pore space, you would like to know what is a surface area also. So, modern-day porosimetry gives you the advantage of obtaining the particle sizes also surface area also by mathematical modelling and the total volume of the pores which are present in the system. These are the catalysts.

So, catalysts are the one which has active surfaces, and they give a chance for the species chemical species to come and get parked on them. So, these are mostly activated particles, montmorillonite itself is an activated particle or I can create a sort of a zeolite by treating quartz with sodium hydroxide at elevated temperature. So, this might get converted into a material of higher cation exchange capacity.

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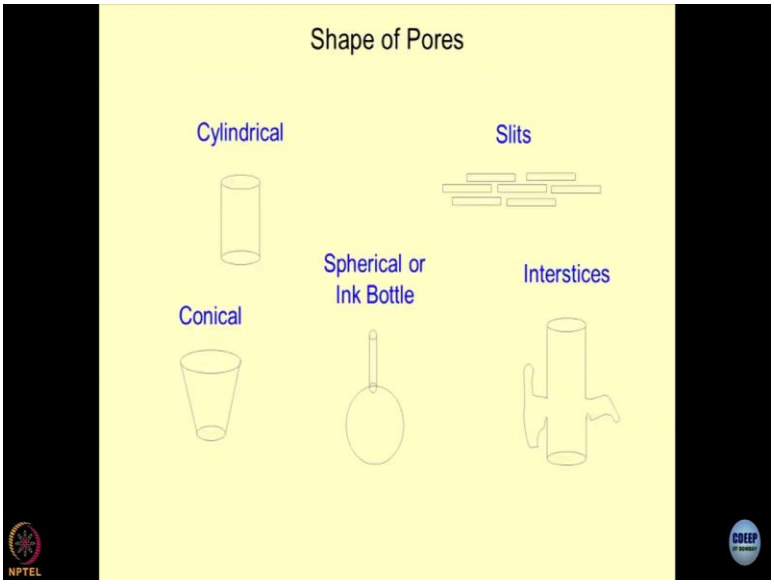

These are the different shapes of the pores which are normally used in the analysis. So, there could be the pores which could be modelled as cylinders of certain diameter and length, slits like the dispersed structure. So, these are also the pores in this slit form, we could have conical pores, both sides the diameter of the pore might not be same funnel sort of a thing, this could be the connection to the outside environment, and this would be inside the material or vice versa, this could be inside the material, and this could be outside the environment.

So, I hope you understand the consequences of this type of arrangement in the soil mass. Similarly, we have ink bottle effects also or ink bottle types of pores also. So, there is a small cylindrical pore which is connected to a voluminous pore, and this becomes a typical ink bottle. We have interstices also the pores which are connected with each other. So, these are the types of pores which are normally model in porosimetry.

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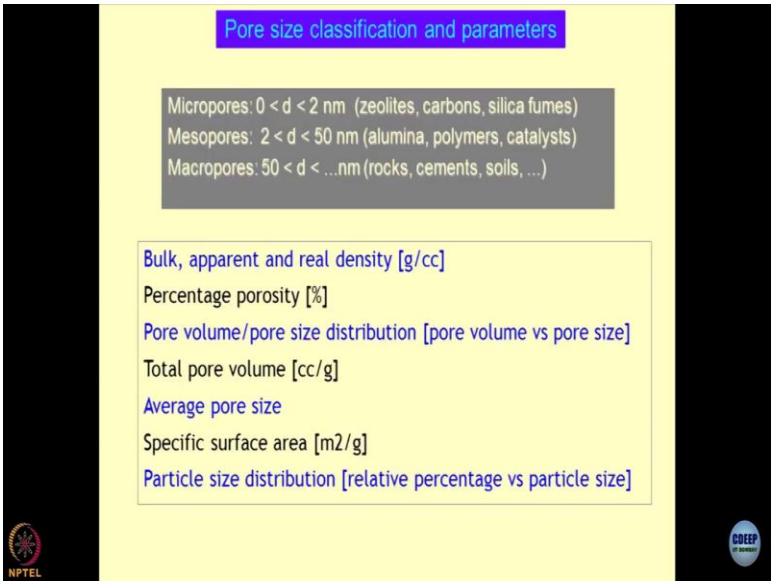

When we talk about the pore size the in geomaterials, we use this classification scheme, Micro Meso and macropores. So, as the name suggests, the micropores are going to be the smallest ones, then we have in between, and the macropores are the big pores. So, up to the 50 nanometers and more than that, these are the micropores, and the smallest pores are less than 2 nanometers. So, most of zeolites carbon silica fumes which you have studied would fall under the category of micropores.

And different types of alumina, polymers, catalyst which industrialists are using. They fall under the category of mesopores, and the macropores would be different types of soils, cement and rocks. So, when we do pore size classification and characterization, we normally talk about the bulk apparent in the real density of the geomaterials. I hope you can realize that I am using three terms in the form of the density bulk, apparent and real densities.

So, you have to use different techniques to differentiate the types of pores which are present in the system. And I can hope you can realize that bulk is going to be macroscopic in nature and apparent is the one which is due to the presence of the voids which have a lot of air into them and the real would be something which could be skeleton. So, you have to differentiate between these types of the density of the geomaterials to do complete modelling. Then we talk about the percentage porosity; we talk about the pore volume, pore size distribution analysis.

Total pore volume, average pore size, specific surface area and particle size distribution. Fortunately, in today's world, the type of software which we have, these things can be done in a fraction of time very quickly and easily. And most of these things are statistical in nature. And you can do a very comprehensive analysis of the sample which you are studying for its pore structure.