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Lecture - 24 Geomaterial characterization -4 (Mercury Intrusive Porosimeter)

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IIT Bombay	Mercury Intrusion Porosimetry (MIP)
	aterials are composed of wide range of particle sizes and nd are porous in nature.
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Ø	Inter-connected Passing Dead end
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Well, let us discuss the second methodology which is MIP (Mercury Intrusion Porosimetry). So, why you need to conduct MIP? What are the requirements? So, basic understanding is on the geomaterials which are compose of wide range of particles, their sizes, shapes and the pores or the material is porous and media. So, this were the knowledge of pore structure of these materials is important, as it can give insight in to both microstructure and the performance.

See there was a time and people used to talk about particle size distribution. Now particle size distribution is very well understood and its limitations are also very well known. So, present day geomechanics you know entails MIP analysis to understand, what type of pores are present in the soil mass and what is their distribution in terms of diameter radius and so on and frequency.

So, rather than measuring the porosity it becomes more informative, if the manner in which volume is distributed with respect to the pore size. So, lot of people you will find a working in this area, where they are trying to develop equivalent seepage models for soil

mass; where there is a very serious you know doubt or question being raised whether your has not personally equation is correct whether Darcy's law is correct. So, these types of studies people are trying to do. Because this equation do not consider the overall porosity of the system and in the subsequent slides I will try to show you, that when we talk about the porosity, I think we are only taking in to account hardly 20 to 40% of the porosity of the system, which you call as the bulk porosities now the total porosity.

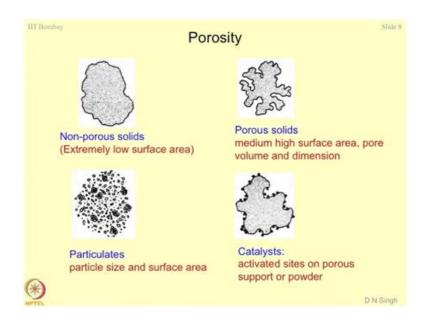
Now, for that matter if you look a grain of the soil and the pores how they are you know associated with each other. So, there could be some pores which are closed, there could be some pores which are dead ends. So, it's just like driving a vehicle in a lane which ultimately ends up in a dead end. So, you think of water molecules entering the system, but then there has no way out to come out of the system. So, in what way the overall hydraulic conductivity is going to we are affected? If you have not included dead ends which are present in the pores of the soil mass.

Similarly, for that matter you can see some of the pores are interconnected. So, the moment water comes over here gets distributed in two parts and both of them end up in dead ends. So, those who are working in to the micromechanics of the systems, they required these types of analysis much more. And this is where the energy concepts are becoming very important in geomechanics because using the energy concepts particularly the law of thermodynamics, you can analyze what is happening inside the porous material. Now most of the time we talk about the pores which are through and through like these are the pores, you know these are the passing pores.

So, any water which enters in to this simply comes out of the system and we call it as a short-circuiting affect. That means, the water as tendency to short circuit the sample itself it is not entering to the sample it is passing through the pore through and through; without taking into account the complete porosity of the material. So, this is where most of the time people have given a thought that how total porosity of the system can be measured and that is where mercury intrusion porosimetry becomes quite important.

In western countries they do not use it; because of the reason that it deals with mercury and their too much scared of in a mercury as a poisonous and toxic material, but anyway we continue our study using these things.

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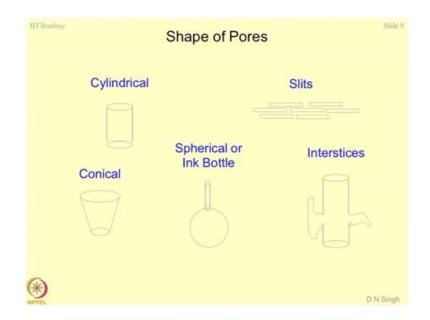


So, whatever porosity is you have till now talked about, look at this grain; there another grain like this of the sand or the clay or whatever there is a agglomeration of the grains suspensions and this another grain like this. So, if you want to understand what is meant by porosity, the first type of the solid is known as a non-porous solid. It will not have any tendency towards fluid no attraction clear? Nothing can pass through this.

So, this is extremely lower surface area. Most of the sands will show you this type of a porosity this is where we talk about their surface porosity. How about this system these are the porous solids, they have high surface high pore volume and high dimensions. Because of all the protrusions and interstices which are present in the system. Now this is a particulate structure where you have particle size and the surface area which has quiet high and this is a very interesting type of a porous material, which is sometimes also named as catalyst. So, on the particle you will see that lot of ions cations or the foreign body they have tendency to get parked.

Now, this process we will discuss later on under sorption desorption capacities of the materials. So, basically these are the activated sites on porous systems which are normally present in powders or very fine-grained materials. So, we talk about the porosity the whole idea is to distinguish between this to; this to; this to this type of grains which are present in the soil mass.

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These are the shapes of the pores which are present in the soils; they are cylindrical in nature; they are the slits. So, a material will pass through like this it will come over here again pass through like this and so on.

So, it's a slit structure conical pores a spherical or ink bottle type of pores you have. So, enough permission can take place through this ultimately there is a dead end in this form and everything gets accumulated over here. And sometimes you have interstices interlinking of different types of pores. So, these are the basic models which people have come up with when they talk about the porosity of the system. Now how do identify a pore is a million-dollar question. So, that is again is the very intricate thing how would you differentiate between these types of pores?

The process of MIP and its results are required to be discussed from that basically we can understand which are the of course.

One of the examples I will show you how to differentiate between the two types of pores at least; any questions doubts? That is what I said these are the assumptions, these are the people have assume that these type of pores will exist and they have done mathematical modelling and they are try to corroborate the results from the experimental results and then they show whether this type of system as cylindrical pores or slit type of pores or ink bottle type of pores or interstices and so on. Lot of research is going on in this area, you will not believe this, but this is the micro analysis of the soil samples and for understanding the interaction which we have been talking about, you have to understand this type of phenomenon and the mechanisms then only you can talk about how the contaminants are getting you know interacted with the soil mass.

So, when you start talking about interaction, then you have to go into the micro mechanism.

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	Micropores: 0 < d < 2 nm (zeolites, carbons, silica fumes)
	Mesopores: 2 < d < 50 nm (alumina, polymers, catalysts)
	Macropores: 50 < d <nm (rocks,="" cements,="" soils,)<="" th=""></nm>
B	ulk, apparent and real density [g/cc]
P	ercentage porosity [%]
P	ore volume/pore size distribution [pore volume vs pore size
Т	otal pore volume [cc/g]
A	verage pore size
S	pecific surface area [m2/g]
P	article size distribution [relative percentage vs particle size

So, roughly if you characterize or if you classify the pore sizes and the parameters which you can obtain from this type of analysis, there are three types of pores; micropores, mesopores and macropores. I think now you can understand in which zone you normally talk about when you do classical geomechanics, these are all in nanometers range. So, when you talk about micropores there are less than 2 nm, most of the zeolites carbon silica fumes they will have these types of pores.

Mesopores there are from 2- 50 nm, alumina, polymers, catalyst they will exhibit these types of pores. And macropores are the pores which normally you talk about when you do your porosity, this to obtain the porosity and that is what I say you only you know talk about one third or one fourth of the porosity of the total system ignoring the micro porosity and meso porosity. For which you require either helium gas or mercury so, that under pressure these fluids will enter and they will reach up to these pores water molecule cannot reach up to these pores.

So, that is the difference why you require some non wetting fluids like mercury and helium gas to do pore structure analysis. Now these are the parameters which normally one will look for from the analysis, bulk property, apparent and real density, percentage porosity, pore volume, pore size distribution that is the pore volume versus pore size. So, rather than particle size pore size is more important this is what I am trying to emphasize over here. Is the pore volume versus pore size not the particle size the total pore volume in total volume divided by per unit weight of the material. Average pore size, specific surface area of the material and particle size distribution that is relative percentage versus particle size.

So, this information you should be getting from a typical mercury intrusion porosimetry. Anything else? Sneha which you can think of getting from these tests or this is a complete system in your hydraulic conductivity models what are the parameters you have used to define k hydraulic conductivity? Normally you say k is proportional to e/(1+e) or $e^2/(1+e)$ or $e^3/(1+e)$, but there are lot of coefficients with get multiplied to this what are those coefficients.

Shape constant.

Shape factor tortuosity factor.

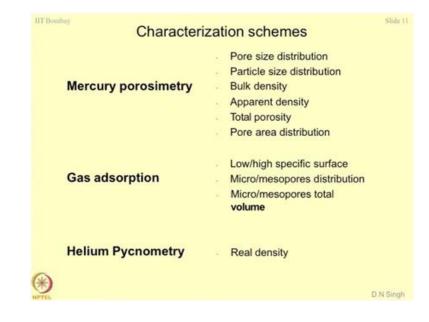
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Sorry.

Diameter (Refer Time: 11:22).

Diameter that is for coarse grained materials yes. So, shape factor tortuosity these are the two important things. So, we have been always assuming or ignoring these factors, we never talk about shape factor and tortuosity. But slowly and slowly when you go to the modelling of contaminant transport, you will notice that the tortuosity factor becomes much more important. So, you can still use these techniques to obtain shape factor c parameter and tau parameter these are tortuosity. I hope you understand what is meant by tortuosity factor that is effective length divided by physical length of the sample is square of that.

So, that is why tortuosity factor could be much much higher than one and hence your hydraulic conductivity gets affected because of tortuosity of the particles.

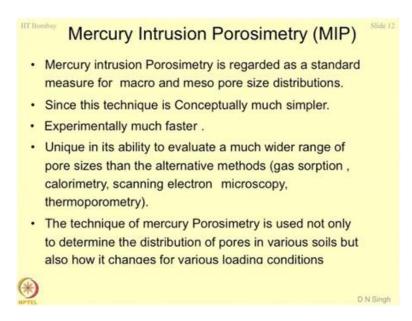


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So, the lot of scope of doing research in this area, then comes the characterization schemes and here I am going to introduce a bit of gas adsorption, because latter half of the lecture is going to be associated with the gas adsorption techniques for finding out surface area and so on.

So, MIP basically gives you pore size distribution, particle size distribution, bulk density, apparent density, total porosity pore area distribution. You require gas adsorption also sometimes because in finding out let us say surface properties. So, if you are interested in finding on low high specific surface area, then gas adsorption is a good technique. Micro mesopore distribution; that means, further differentiation between the pores which are present in the system micro mesopore which are in the total volume and of course, helium gas pycnometry which will give you the real density of the system is this part clear.

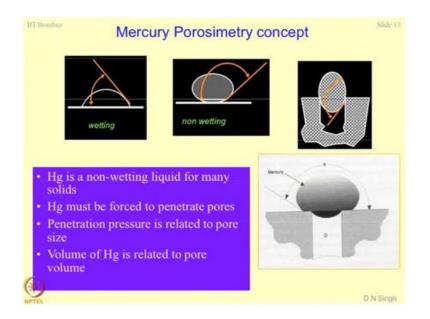
So, we have discuss about MIP quiet in detail helium gas pycnometry instrument you must have seen in the lab, where you can pass helium gas and you can get the real density; about gas adsorption I will show you what is the application and how these techniques are used for characterization of the geomaterials.



A bit of the MIP details. So, mercury intrusion porosimetry is regarded as a standard method for macro and mesopore distributions analysis and since this technique is conceptually much simpler, it is experimentally much faster. And this is unique because of its ability to evaluate a much wider range of pore sizes than the alternate methods like gas sorption, calorimetry scanning electron microscope and thermoporometry. How much heat gets diffused in the soil mass from one place to another place this is what is known as thermoporometry.

And this technique is used not only to determine the distribution of pores and various materials of the soils, but it is also to find out the changes which a system undergoes during loading conditions. One example is that compaction process. So, from dry to wet of optimum when you compact the soil, how your pore structure is changing can be captured by this technique.

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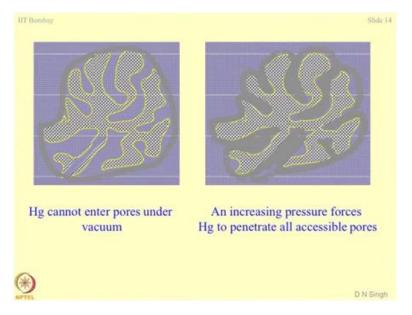
This is the basic concept of porosimetry. A typical wetting fluid will show you this type of menisci and the angle or the tangent between the surface and the menisci will be theta value which is going to be lesser than a non-wetting fluid.

So, in case of non-wetting fluid because of the surface tension this is how the fluid will set each drop of the fluid. Now using this concept, I can pass it through a pore of size d by a plane certain pressure without breaking the drop clear. So, if you think that this is a tube which is nothing, but your capillary tube you have done this analysis that if this is a surface tension pores. So, t times multiplied by cos theta in to the area cross section is the uplift pores t sin theta component gets nullified and these pores is lifting the fluid in the capillary, that is the volume multiplied by density. So, from there you can get the equation which governs the law of mercury porosimetry, this is known as Washburn equation I will show you slightly later.

So, the characteristics which you use are mercury the nonwetting fluid for many solids and it is force to penetrate in to the pores and penetration pressure is related to the pore size. So, this is the pore size, the amount of pressure which you are applying from outside their correlated with each other; that means, a certain pressure will take care of a certain pore size. So, I will show you a typical MIP result which will be self explanatory and to show you how this type of analysis can be done and you can achieve some conclusive results. Another interesting thing is that the volume of mercury which is going in to the pore is directly related to the pore volume.

So, here we are showing a pore in this way, this pore could be cylindrical ink bottle type slit type or anything as per the previous models. Now this is what happens when you are trying to intrude mercury in to a pore. Just imagine as if your parking a mercury droplet in to a pore by applying some external pressure and the condition is that this mercury drop should not break clear due to the external pressure. So, it's a basically its energy between the; between the pore and the fluid which is trying to enter into it.

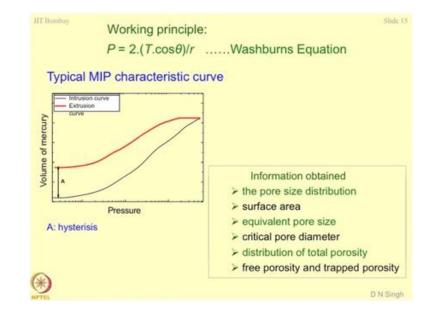
So, the more pressure you apply here what will happen? Either the pores will change or the droplet will change. So, this type of contradictions will come sometimes when you do this type of analysis. So, I would like to show you one figure.



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Now, this is the basic model which is used to understand a wetting and a non-wetting fluid. If you have a system of particles where the pores are you know concealed by a layer of the liquid what happens? Mercury will not be able to enter into this however there could be a system where if you apply more pressure what will happen? This mercury has to enter into this.

So, this is a basic concept of choosing a pressure and how this pressure is going to result in intrusion of mercury into the pores of the system. So, basically this becomes a interesting phenomena, what pressure should be applied for a given pores so, that these pores of a certain diameter get buildup is this ok.



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So, this equation you can derive easily by the mass balance that is the surface tension T $\cos\theta$, r is the radius of the pore or the capillary and P happens to the pressure. So, this is how you are computed suction pressure also in the soil mass by using the same equation in this and θ is a contact angle now this is what is known as Washburn equation.

So, T is known for mercury and θ is known for a soil mercury system 130 to 140 degree and T is 0.48 dynes per centimeter as suppose. What it leads to is a typical mercury intrusion porosimetry characteristic curve. So, if you look at this curve what you notice is on the y axis is volume of mercury, which is getting entered in to the system or coming out of the system with respect to pressure on the x axis. So, how we read this graph is, if you increase the pressure what will happen? In mercury it has a tendency to go into the pores. So, this becomes an intrusion curve.

So, the black one is intrusion curve as you increase the pressure applied the more and more volume of mercury goes in to the pores of the soil mass. So, this way you can complete all the pores by applying high very high pressure. Now what is going to happen when mercury is entering in to the pores of the soil mass can you in speculate something? It may break the pores itself. So, there could be a collapse of pores. So, now, this is what you are going to see here is just like a consolidation graph you know where

this is known as intrusion curve, the mercury goes in to the pores and if I retreat back the mercury by applying suction. So, what is the graph which I get this is what is known as extrusion graph. That means, if I can extrude the mercury which as entered in to the system by decreasing the pressure or by applying some vacuum, this is where you end up. So, the sum up volume of the mercury remains and the soil mass this could be because of the distraction of the pores and this phenomenon is normally known as hysteresis.

Now hysteresis becoming a very big question mark in geomechanics. Everywhere we talk about hysteresis if you remember in electromagnetic courses their due to talk about hysteresis and based on the hysteresis, we have classified the material. You agree or no a material with stores more of the magnetism has more hysteresis of this concept can be utilized in geomechanics also in characterizing the materials and differentiating between two types of materials.

And the type of pore structure which is existing in the system; is this part ok? So, this is what Sneha is trying to study and she will be presenting a seminar on wetting and drain curve; where she will be talking about hysteresis associated with wetting and drain, now that again in the capillary action you agree or no that is what you are trying to study. That is what Seema is trying to answer in her phases when she talks about soil water characteristic curves during wetting and drain. So, why their different or why they are similar?

So, these are three examples of hysteresis which normally we talk about. Now NC to OC responds in classical geomechanics is also nothing, but hysteresis. So, whole the define hysteresis? Hysteresis nothing, but the amount of energy with gets stored in to the system.

System here what is happening? These amount of mercury which is getting stored in to the pores. So, there must be some pores which is capturing all the mercury in to the pores or this mercury cannot come out because of several reasons one of the reasons could be collapse of capillaries.

So, Sneha this is a challenge, later on maybe when I talk about hysteresis in trying in shrinking phenomena, their you can notice that your proctor compaction curve in wetting cycle will be different than the proctor compaction curve which you obtain in the drying

cycle. So, these are the you know affects of environment which are causing drain and wetting of the material. When moisture goes in to it or comes out of it. So, these types of tests which we are discussing like MIP are nothing, but a good simulation of a natural phenomena in terms of quantification of its pore structure and the alteration in the pore structure because of some process. Is this part clear? If pores were perfectly elastic, what would have happened?

All the mercury would have.

Come out.

Come out.

Exactly, so, the red line and the black line would have, super or super impose perfectly over region that is right. So, no loss of mercury no loss of pores and so, on.

Sir, but what is the minerology effect (Refer Time: 24:03).

That is what I said the way you characterize your ferromagnetic material, non ferrous materials, non ferromagnetic materials, what are the differentiation is just based on the amount of magnetic energy with this store.

Exactly that is what we; that is what we are doing that is what we are trying to do and that was the reason of telling you that the consolation theory should be revisited by using energy concepts just by applying mechanical loads you cannot find out the volumetric deformation to a precise limit; you are ignoring lot of mechanisms which are going on in the sample and you are just measuring something which is not very accurate, I think now you can correlated better. So, similarly somebody may ask you to develop a model based on energy model for stress-strain relationship of materials, why you get peaks in OC material why you do not get peak in NC material why do get a peak in heavily compacted sands.

And you do not get any peak at the time of sharing in loose sands. So, everywhere you are finding this is the packing of the grains which is getting affected by the particle arrangement. So, lot of things are going on inside the particle which we have not capturing to the minutest details. And that is where this type of thinking and philosophy is you know is going to be quite helpful in answering the mechanisms which are very

close to the nature good. So, since that today their followed things is this any other suggestion no doubt.

One more additional this thing means about this hysteresis another reason could be you know this entrapped mercury it is because of the pore shapes which sir as already shown for example, that ink bottled type. So, while intruding, it will get entered, but while extruding it is not possible to come out all at mercury because it got entrapped in to that bottle shaped. So, that could be another reason means even if the pore structure is intact, but because of the pore shapes it is possible that mercury will get entrapped and it is not possible to come out.

My question is how can we intrude (Refer Time: 26:19).

Or in another word you mean what you can say its soil contemnor interaction how it going to affect the pore structure.

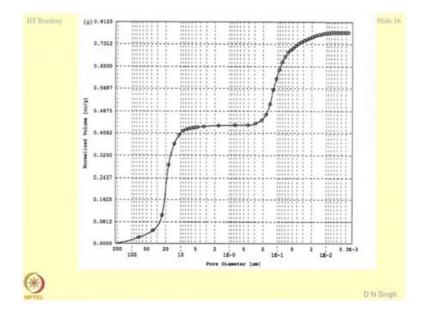
Correct.

That we can understand from this.

The degradation of the pore porous media because of interaction it becomes a reverse problem you got it? It's a very good thought which you are giving that is right. So, you can see the after effect and then you can back trace the effect this is also known as back analysis. So, degradation of the material materials responds to external disturbances you can study easily. I have included one slide what he was talking about an I hope it should be clear I showed that slide. So, what is the information which you get from this type of a relationship? So, basically what you are doing here is you are developing a incidentally the day you look at it is, it's nothing, but your consolidation curve is it not your pressure sigma prime versus wide ratio e versus sigma prime curve.

So, e is nothing, but there you talk about the compression of the grains sorry compression of the pore structure wide ratio wide ratio keeps on changing. So, these are the same graph. So, what information will get from is this? The pore size distribution surface area equivalent pore size, critical pore diameter distribution of total porosity and free porosity and their trapped porosity, this trapped porosities Suchith was discussing.

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Now, this is a typical intrusion curve which we got for one of the samples which we had analyzed. So, where you have pore diameter versus applied volume normalized volume. So, the more the pore diameter what happens? More and more volume of mercury is going to get intruded into the system. Now could you answer this question that you find here one bench, what is the reason for this bench formation? That means, the normalized volume does not increase with the pore diameters clear mercury is a noncomprisable fluid.

So, they will not be any time lag it shows the porous structure property that the pore diameter of this range is quiet prominent. So, it just like free flow of fluid in to the systems. So, with a with no even increment the pressure, the entire mercury goes and sits in to the pores spaces, but then I just may wait a minute please and then if you want to further overcome the resistance offered by the pores you have to apply more pressure. So, truly speaking as the pressure increases you know the normalized volume keeps on increasing which is being mapped over the pore diameter.

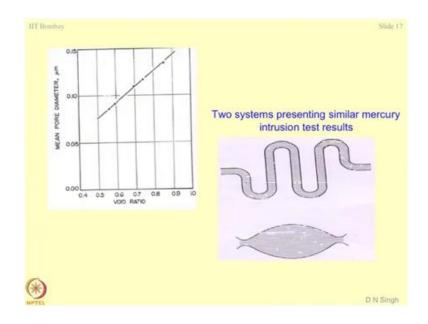
So, this is a good way to see what type of distribution of the pores you have in the material and how easily the pores are getting filled up with the mercury because of certain pressure. So, this type of diagnosis you may do by looking at these types of graphs yeah what you are saying.

Sir, why if the volume is not increasing if that pore size increasing?

Because of the type of the pores; that means, from pore sizes of let us say 22,.2, the most of the resistance is not coming to the picture. So, these are the pore which are good enough for mercury to flow just like this very easily you do not require any external pressure. So, in other words there is the predominant of the pores of this size in the sample this is the resistance offered by the system and this is the resistance offered by the system. So, you can very easily make out that this is the range in which the pores are present in the soil mass or a porous system.

So, this is how you can diagnose what type of pores and their frequency and their magnitude and their diameters are present in the system. Present day MIPs have software which are included in them. So, they analyze everything for you based on the curves which normally you get by doing an experiment those of you who are interested learning MIP, they can contact Suchith and whenever he is doing his experiment next time you can come and sit with him it takes time. So, you please fix up with him and you can demonstrate to them how it works.

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Now, this is another interesting relationship which we will get out of this exercise, mean code diameter as a function of void ratio all right. So, as the void ratio increases the mean code diameter also increases yes understood. So, but this void ratio is the total void ratio which we are talking about which includes all sets of pores, micro meso, macro

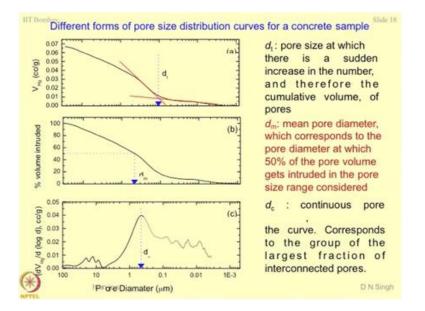
which otherwise you would not be able to obtain; that means, the material and the grain porosity also you are considering in to the analysis.

Now this answers your second part of the question when you are talking that how these studies can be implied for soil-contaminant interaction. You will notice if the subsequent lectures while sorption desorption takes place, there are lot of parking of the cations which is taking place on the grains of the clays. So, unless you know exactly how much is the surface area which is expose to the environment or the contaminant, you will not able to estimate how much interaction is going to occur.

Now, this is what actually Suchith was trying to explain, you may have a system where the mercury gets entrapped inside and there are both the ends which are choked. So, let us orifice on both the ends, but more cross section which is available for mercury to get filled up. Now other way of understanding this would be there is no difference between this type of pore and this type of pore. So, truly speaking this is the limitation of MIP you cannot really mapped the pore structure; because this is equivalent to this all the mercury which is entrapped in this portion can be you know drawn an equivalent with this type of a analogy.

So, two systems which will present almost the same responds, but it's very difficult to differentiate between two type of pores. You have to think of something new what differentiating between these two types of pores. For civil engineers this may not be very important at this moment, but those who are working in synthetic polymers, synthetic Zeolites, catalysts for then these studies are very very important particularly those who are I came across a sponge iron sample, where people who wanted to do MIP. I hope you can appreciate why MIP should be done on a sponge iron.

How much amount of energy is required to melt it for that pore structure required to be study it and lot of resins, polymers, catalysts which are being fabricated by the industries required this type of studies and they contact us for doing this test for them.



So, more interpretation on the results which you get from mercury intrusion porosimetry; with this is the complete tomography of the sample you cannot come across things better than this. This work was done by another PhD scholar doctor Bibhuti Das and we studied here the concrete specimens. Like in your particle size distribution curve you use lot of terms as c_c , c_u , D_{15} , D_{85} , D_{30} , D_{60} is it not what for or what purpose? So, that you can characterize the material better where do you use cc and cu terms in classification of the soil coarse grained materials.

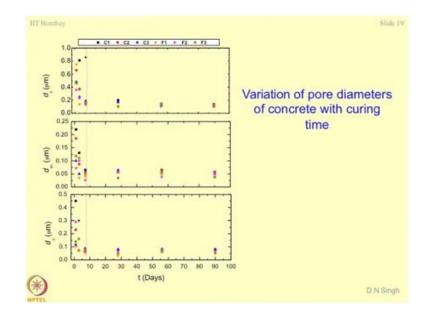
For fine grained materials you have another classification scheme, liquid limit, plastic limit plasticity index atterberg limits and so on and then drawing the a line. So, the question is that after doing MIP, how I am going to differentiate the materials? How I am going to use the parameters to differentiate between one material another material? So, if you look at the first relationship which is the volume of mercury which is intruded in the sample as a function of the pore diameter all right.

So, as the pore diameter increases, what you will notice is there the volume of mercury drops down. Now if you do a bit of construction now this type of construction you have done to find out the creep and t_{90} if you remember 90 % consolidation in the soils. So, the mechanism remains same the pore diameter versus volume of mercury graph will give you a parameter which is known as dt.

So, dt is nothing, but this pore size you know there will be a sudden increase in number and the cumulative volume of the pores. So, this parameter becomes very important in characterizing the geomaterials. If you plot percentage volume intruded of the mercury versus pore diameter, this is something known as d pore diameter d_m which corresponds to 50 % of the pore volume. So, if you consider 50 % of the pore volume if you obtain d m this is becomes another parameter which can be used for characterizing the geomaterials. And the last parameter is dc this is known as continuous pore diameter, this is what I was talking about in the previous slide that pores which are continuously staggered or continuously present in the system.

So, this is the term which is known as dV_{Hg} that is increase in volume of the mercury divided by differential changes in the log of the pore diameter. So, if you plot this index with respect to pore diameter wherever you get a maxima, now this maxima corresponds to dc. So, this is the continuous pore diameter the maxima of the curve which corresponds to the group of the largest fraction of the inter connected pores. So, this gives to the answer to the point which you are raising and when the pores are inter connected you do not require much pressure to push them from one pore to another pore to another pore to another pore conductivity is very simple. So, based on dt, dm and dc there your equivalent terms to D₅₀, D₃₀, D₆₀ and D₈₅ and D₁₅ and then you can try to see what is happening some material.

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I thought of showing you this relationship which you will appreciate which shows that how curing of the concrete can be checked you know by using these 3 parameters. What do you want concrete to become after 28 days much more durable; that means, the pore sizes should be as small as small possible. So, if you plot dt dm and dc with respect to days of curing, what will notice is as days increase there is a decrease in pore diameter and that is what you wanted clear. So, it's a very simple application of MIP in checking the pore media characteristics clear? So, this is how you can go for synthetic concrete as well by using different type of pozzolanic materials and making a good grade of concrete. In the next class I will discuss the specific surface area determination.