Environmental Geotechnics
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Lecture – 26
Geomaterial characterization – 6 (Specific surface area- II)

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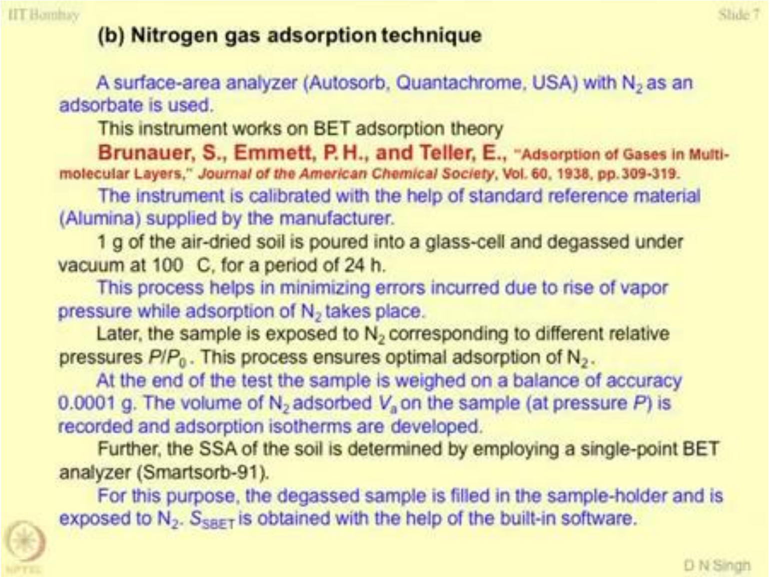

(b) Nitrogen gas adsorption technique

A surface-area analyzer (Autosorb, Quantachrome, USA) with N_2 as an adsorbate is used.

This instrument works on BET adsorption theory

Brunauer, S., Emmett, P. H., and Teller, E., "Adsorption of Gases in Multi-molecular Layers," *Journal of the American Chemical Society*, Vol. 60, 1938, pp. 309-319.

The instrument is calibrated with the help of standard reference material (Alumina) supplied by the manufacturer.

1 g of the air-dried soil is poured into a glass-cell and degassed under vacuum at 100 C, for a period of 24 h.

This process helps in minimizing errors incurred due to rise of vapor pressure while adsorption of N_2 takes place.

Later, the sample is exposed to N_2 corresponding to different relative pressures P/P_0. This process ensures optimal adsorption of N_2.

At the end of the test the sample is weighed on a balance of accuracy 0.0001 g. The volume of N_2 adsorbed V_a on the sample (at pressure P) is recorded and adsorption isotherms are developed.

Further, the SSA of the soil is determined by employing a single-point BET analyzer (Smartsorb-91).

For this purpose, the degassed sample is filled in the sample-holder and is exposed to N_2. S_{SBET} is obtained with the help of the built-in software.

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The second method is nitrogen gas adsorption technique. This is where we use a surface area analyzer which is known as Autosorb which is Quantachrome make, when nitrogen is used as an adsorbent or adsorbate. So, this instrument works on the principle of BET, you have been hearing the name BET several times. So, BET is a combination of three researchers like Bruner Emmett and Teller. So, in the name of these three researchers this method has been named as BET and this has reference you can go through whenever you get time as option of gases in multimolecular layers, journal of American chemical society 1938.

So, most of us were not even born at that time when this method was developed alright. So, this instrument has to be calibrated first with the help of standard reference material. So, normally alumina is used which is supplied by the manufacturers. This is where you use one gram of the air-dried soil is poured in a glass cell and it is degassed under vacuum at 100°C for a period of 24 hours. Now, degassing is nothing, but removing all the vapors which are associated with the sample and why you are removing all the vapors? So, that the nitrogen gas gets a better chance to get adsorbed on the surface of the particles.

This process helps in minimizing the errors which are incurred due to rise of vapour pressure while absorption of nitrogen takes place. Later the sample is exposed to nitrogen gas corresponding to different relative pressures P/P_0.

where P = applied pressure

P_0 = vapour pressure at saturation.

So, this process ensures optimal adsorption of nitrogen. Incidentally some of you may get a chance to work in carbon dioxide sequestration in your post-graduation or whatever after masters.

So, this is where these types of techniques can be utilized for finding out the recharge capacity of the geo materials, when you talk about carbon dioxide sequestration, methane sequestration and you know expansive soils and so on. Kunal you want to add something here with respect to carbon dioxide sequestration?

Student: Yeah.

Right now, the biggest challenge is how do capture the carbon dioxide from the environment and you know tap it and force it into the aquifers so, that environment becomes clean. So, on this lot of people are working particularly in geotechnical engineering, it becomes a multi disciplinary area where people from chemistry background, earth sciences, geologist geotechnical engineers particularly hydro geologist all of them are considering this problem and this is a very good methodology to capture and reduce the carbon dioxide content in the atmosphere.

So, at the end of the test the sample is weighed on a balance of accuracy 0.0001, 0.1 milligram basically and the volume of nitrogen adsorbed, V_a is the sample corresponding to a pressure is recorded and adsorption isotherms are developed where do you use the word isotherm same temperature.

So, here actually adsorption isotherm is a misnomer. It is the adsorption response at a given temperature. So, isotherm corresponds to the response of the system corresponding to a certain temperature or certain ambience conditions. So, the movement you change the ambience conditions your responses are going to be different and there in comparable. So, that is why we use the word as sorption isotherm, adsorption isotherms, gas isotherms, chemical isotherms and so on.

That means, at least one condition is fixed that the temperature remains same pressure remains same. Further the specific surface area of the soil is determined by employing a single point BET analyzer known as smartsorb, this is commercially available equipment and for this purpose the degassed sample is filled in the sample holder and it is exposed to nitrogen.

You will get surface area corresponding to single point BET with the help of the software which is built in the system. It is a very expensive instrument a BET would cost something like 50-60 lakhs, not less than that and a single point BET will cost you not less than 7-8 lakhs, it is ok.

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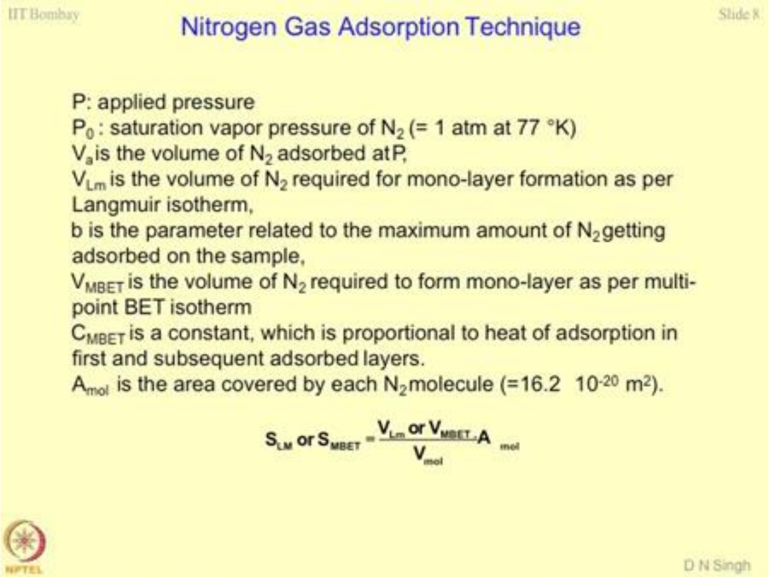

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Nitrogen Gas Adsorption Technique

P: applied pressure
P_0 : saturation vapor pressure of N_2 (= 1 atm at 77 °K)
V_a is the volume of N_2 adsorbed at P,
V_{Lm} is the volume of N_2 required for mono-layer formation as per Langmuir isotherm,
b is the parameter related to the maximum amount of N_2 getting adsorbed on the sample,
V_{MBET} is the volume of N_2 required to form mono-layer as per multi-point BET isotherm
C_{MBET} is a constant, which is proportional to heat of adsorption in first and subsequent adsorbed layers.
A_{mol} is the area covered by each N_2 molecule (=16.2 10^{-20} m^2).

$$S_{LM} \text{ or } S_{MBET} = \frac{V_{Lm} \text{ or } V_{MBET} . A_{mol}}{V_{mol}}$$

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So, if P happens to be the applied pressure; P_0 is the saturation vapour pressure of nitrogen. V_a is the volume of nitrogen adsorbed at a pressure P, we want to measure V_{Lm} is the volume of nitrogen required for mono layer formation. Monolayer is nothing, but the first layer which gets formed under system and it is assumed that they will not be any multiple layer formation on the material as such as per the Langmuir isotherm. Langmuir was the scientist who has given one isotherm. So, in his name the name of the isotherm is Langmuir.

b is the parameter related to maximum amount of nitrogen getting adsorbed in the sample V_{MBET} is the volume of the nitrogen required for mono layer formation using multi point BET isotherm and C_{MBET} is a constant which is proportional to the heat of adsorption in first and subsequent adsorbed layers.

So, Srinivas this where actually we can talk about you know heat which is associated with the interaction and this is where we can define the contaminant soil interaction particularly in the form of heat which comes out of the interaction. And A_{mol} is the area covered by each nitrogen molecule which again remains fixed. So, using this equation that is if you are finding out this surface area from the Langmuir isotherm or from the multi point BET you can use the volume divided by the volume of the total system into the area of mono layer.

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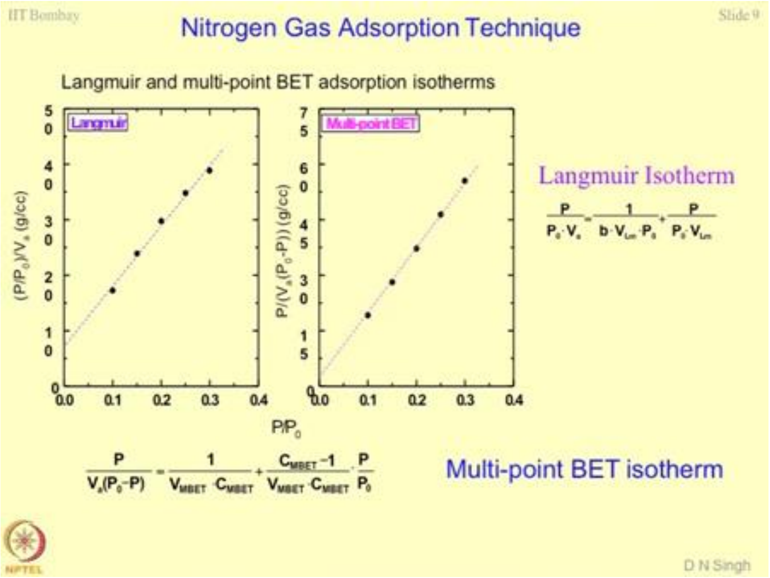

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Nitrogen Gas Adsorption Technique

Langmuir and multi-point BET adsorption isotherms

Langmuir Isotherm

$$\frac{P}{P_0 \cdot V_a} = \frac{1}{b \cdot V_{Lm} \cdot P_0} + \frac{P}{P_0 \cdot V_{Lm}}$$

$$\frac{P}{V_a(P_0 - P)} = \frac{1}{V_{MBET} \cdot C_{MBET}} + \frac{C_{MBET} - 1}{V_{MBET} \cdot C_{MBET}} \cdot \frac{P}{P_0}$$

Multi-point BET isotherm

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What this will give you is a relationship like this. So, these are known as nitrogen gas adsorption isotherms. On y axis you will find the P/P_0 value divided by the total volume which gets off on the system alright and then on the x axis you have P/P_0. So, if you remember the gas equation universal gas equation this is.

Student: PV= nRT.

PV= nRT. So, here what we are doing is we are plotting this in such way that P/P_0 is normalized with the volume with respect to P/P_0. So, truly speaking these to get normalize and what you get is only $1/V_0$. So, that is nothing, but the slope of this line. So, this was the hypothesis which was given by Langmuir and the hypothesis which is normally used in MP BET, these are the forms of the equations:

$P/(P_0.V_a) = 1/(b.\ V_{Lm}.\ P_0) + P/(P_0.V_{Lm})$

You need not to remember all this, this is just to expose you again a what type of analysis is done to get the results, you are not supposed to mug up all this information alright.

But of course, it should remain in your mind somebody ask you yes you can say that I know how to get these values or the used instruments or how the analyze the values.

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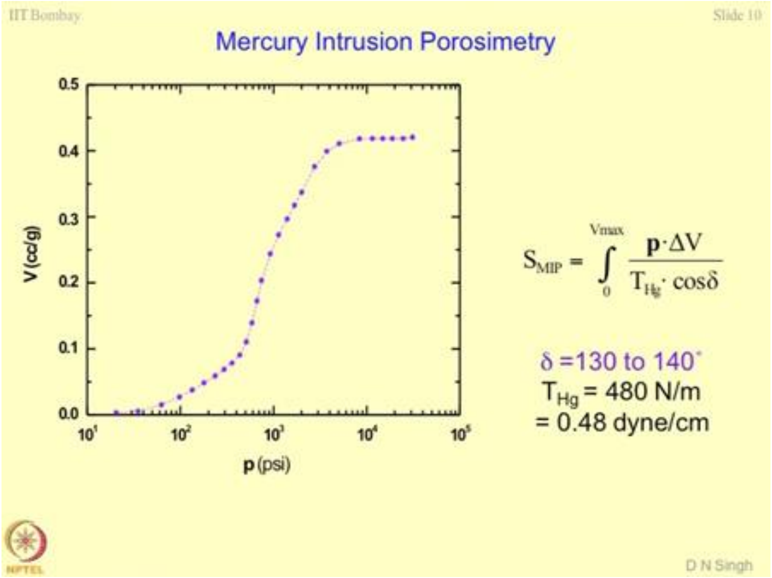

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Mercury Intrusion Porosimetry

$$S_{MIP} = \int_0^{Vmax} \frac{\mathbf{p} \cdot \Delta V}{T_{Hg} \cdot \cos\delta}$$

δ =130 to 140°
T_{Hg} = 480 N/m
= 0.48 dyne/cm

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This is the third method mercury intrusion porosimetry, we were talking about this type of relationship in the previous lecture. The volume of the mercury which is getting intruded into the system as a function of pressure applied pressure. So, you get a typical s curve and what it shows is that the pores are very well distributed and there are all sorts of pores which are present in the system. If you think of a situation where you get a vertical line, what will be meaning of that?

It is not possible first of all; is it not? It is the hypothetical situation; that means, you require one pressure to fill all the volume and your system happens to be highly porous, but this type of is geomaterial is of no use. Now, what is to be done to get the surface area you simply integrate the area which is below this curve. So, how integration will be done? $P.\ \Delta V/(T_{Hg}.\ \text{Cos}\delta)$ from 0 to V max. So, from this point to maximum value of the V volume if I integrate this curve, I will get the surface area which is normally defined as S_{MIP}.

Now my question is this surface area will be total or external or internal; why external?

Student: Mercury is not reacting with.

Mercury is going to surround each particle. So, again this is going to be a sort of a external or total?

Student: (Refer Time: 10:26).

External, only external alright. No, I am just trying to give you an idea that though mercury is intruding into the system, but as he rightly said that is not interacting with material at all. So, it is still remains external surface area. Here normally delta is used as 130 to 140 degree depending upon the soil or concrete technically when you deal with concrete the delta value is 140 degree and for soils it is 130 degree. This is the surface tension of mercury 480 Newton’s per meter or 0.48 dynes per centimetre.

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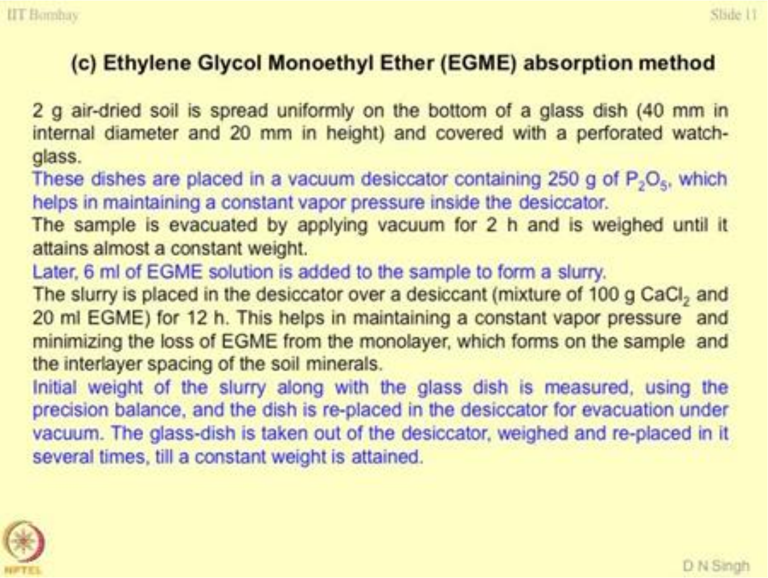

(c) Ethylene Glycol Monoethyl Ether (EGME) absorption method

2 g air-dried soil is spread uniformly on the bottom of a glass dish (40 mm in internal diameter and 20 mm in height) and covered with a perforated watch-glass.

These dishes are placed in a vacuum desiccator containing 250 g of P_2O_5, which helps in maintaining a constant vapor pressure inside the desiccator.

The sample is evacuated by applying vacuum for 2 h and is weighed until it attains almost a constant weight.

Later, 6 ml of EGME solution is added to the sample to form a slurry.

The slurry is placed in the desiccator over a desiccant (mixture of 100 g $CaCl_2$ and 20 ml EGME) for 12 h. This helps in maintaining a constant vapor pressure and minimizing the loss of EGME from the monolayer, which forms on the sample and the interlayer spacing of the soil minerals.

Initial weight of the slurry along with the glass dish is measured, using the precision balance, and the dish is re-placed in the desiccator for evacuation under vacuum. The glass-dish is taken out of the desiccator, weighed and re-placed in it several times, till a constant weight is attained.

Now, comes the method which is known as Ethylene Glycol Monoethyl Ether EGME. This is a very interesting method, 2-gram air dried soil is spread uniformly on the bottom of a glass dish and the dimension of glass dish are 40 mL internal diameter and approximately 20 mL in height.

And this is covered with a perforated watch glass. So, that you can observe what is happening inside. These dishes are placed in a vacuum desiccator containing 250 grams of phosphorus pentoxide, which helps in maintaining a constant vapour pressure inside the desiccator. The sample is evacuated by applying vacuum for 2 hours and is weighed until it attains almost a constant weight. This was the philosophy which we developed to see how evaporation affects you know the fundamental properties of the clay surfaces. Later 6 mL of EGME solution is added to the sample to form a slurry. So, what you are doing is you are allowing soil to interact with a dye.

So, this is the controlled material which is reacting with this soil mass and this slurry is placed in the desiccator over a desiccant normally calcium chloride is used as a desiccant, you must have seen in the laboratory there in a conical flask we have calcium chloride cubes calcium chloride is nothing, but dehydrant for 12 hours and this helps in maintaining a constant vapour pressure and minimizing the loss of EGME from the monolayer, which forms on the sample and the interlayer spacing of the soil minerals.

I have used this word intern intentionally please go through this sentence again and let me know what type of surface area this method will give you. This helps in maintaining a constant vapor pressure and minimizing the loss of EGME from the monolayer.

So, this is a total surface area; that means, the internal as well as the external which forms on the sample and the interlayer spacing of the soil members. So, the beauty of this method is this will give you the total surface area of the soil particles. Initial weight of the slurry along with the glass dish is measured using the precision balance and the dishes replaced in the desiccator for evacuation under vacuum. Evacuation is the word which is normally used to degas the system what take out whatever has been captured by the soil particles. The glass dish is taken out of the desiccator, you keep on weighing it replacing it unless the weighed becomes constant.

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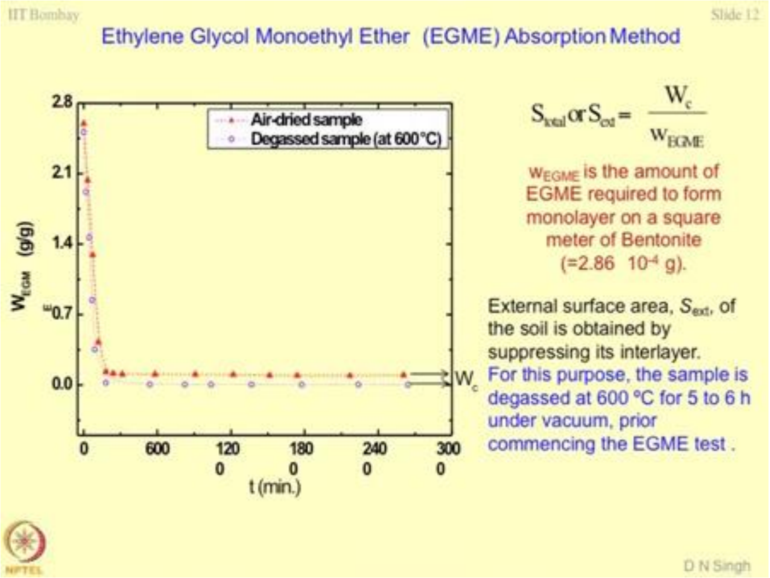

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Ethylene Glycol Monoethyl Ether (EGME) Absorption Method

$$S_{total} \text{ or } S_{ext} = \frac{W_c}{W_{EGME}}$$

W_{EGME} is the amount of EGME required to form monolayer on a square meter of Bentonite (=2.86 10^{-4} g).

External surface area, S_{ext}, of the soil is obtained by suppressing its interlayer.

For this purpose, the sample is degassed at 600 °C for 5 to 6 h under vacuum, prior commencing the EGME test .

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So, this is a type of result which you will be getting. So, typical response of soil which is being treated with EGME, on y axis you will have the weighed with respect to time. So, this is the initial weight of the system and then as time increases what happens? This weight keeps on dropping and then ultimately you end up with W c.

So, here we do two type of experiments; the first experiments done in air dried samples another one is done at the degas samples. Why because, we want to differentiate between the monolayer which has been formed and enter layer between the particle atoms. So, this way we can get both internal surface area as well as the external surface area. Is this part clear? It is a typical drying curve under vacuum suction. If the same analysis has been doing for the soils, you would have found some cracking taking place with respect to time, the weight of the system decreases.

So, this is where we use S total or S external which is nothing, but the weight C which is the residual weight of the sample divided by the total weight of EGME which is used. And this weight of EGME which is used to form a mono layer on a square meter of bentonite is given as 2.86× 10^{-4} grams. So, you can obtain external surface area of the soil by suppressing its interlayer.

How this suppression is being done? This suppression has been done in this form when you have degassing being done and the air drying of the sample, for this purpose the sample is degassed at 600°C for 5 to 6 hours under vacuum prior to commencing EGME test, alright. So, this will give you the surface area depending upon which method you are using either air drying or degassing of the sample? So, you can get S_{total} or S_{ext} depending upon what you have and the minus of the 2 will give you surface area which is internal.

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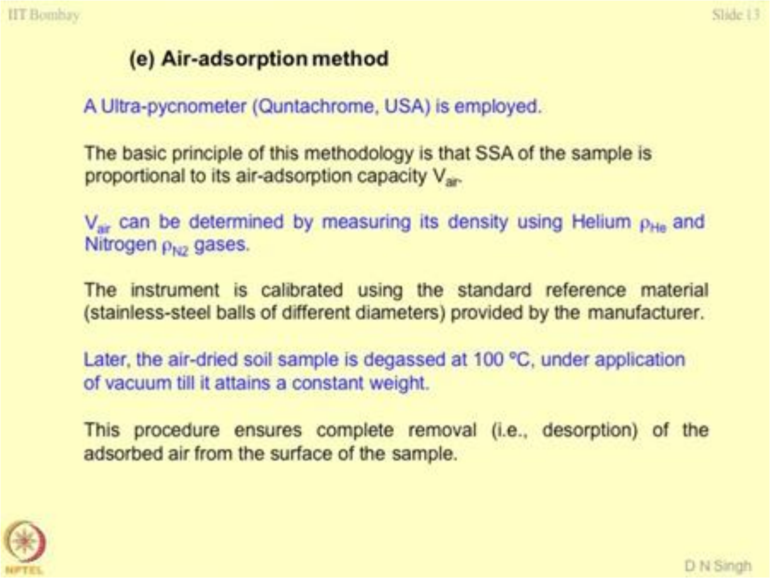

(e) Air-adsorption method

A Ultra-pycnometer (Quntachrome, USA) is employed.

The basic principle of this methodology is that SSA of the sample is proportional to its air-adsorption capacity V_{air}.

V_{air} can be determined by measuring its density using Helium ρ_{He} and Nitrogen ρ_{N2} gases.

The instrument is calibrated using the standard reference material (stainless-steel balls of different diameters) provided by the manufacturer.

Later, the air-dried soil sample is degassed at 100 °C, under application of vacuum till it attains a constant weight.

This procedure ensures complete removal (i.e., desorption) of the adsorbed air from the surface of the sample.

Let us talk about the air adsorption method, let it be a simple method where you can use ultra-pycnometer which is again manufactured by Quntachrome which is available in our laboratory. The basic principle of this methodology is that the specific surface area of the sample is proportional to its air adsorption capacity that is the volume of the air which is getting adsorbed on to the surface and V_{air} can be determined by measuring its density using helium gas and nitrogen gas.

So, you have to use two gases helium and nitrogen. The contrast between the two densities will give you some parameter which multiplied by another parameter will give you the surface area. So, this instrument is calibrated using the standard reference material and, in this case, the standard material happens to be stainless steel balls of different diameters.

You might have seen when you step the lab at the calibration is done with the help of steel balls. Why it is so? Why do you steel balls; any guess? It is the smoothest surface; is it not? So, on a smooth to smooth surface there should not be any adsorption of gases.

You take any rough surface where the adsorption will be more than this. So, this becomes your reference mark where adsorption value is going to be almost nil.

Later the air-dried sample is degassed at 100°C under application of vacuum till it attains a constant weight. Of course, this facility is available in a techno meter itself you can degas the system there itself. This procedure ensures complete removal that is desorption of the adsorbed air from the surface of the sample. Let me ask you a question, what should be the basic function of a deodorant? Why do you use powder talcum, what they what they should do? Yes, what smell they should adsorb within themselves correct.

So, if you want to test a good deodorant a good powder which is used for cosmetics, you have to do these techniques, that is why these techniques are very popular where people who are doing chemical engineering. I mean this is their bread and butter they have to live with these things or everything. I do not know whether you have seen any plant where the nail polishes have been done. You should go to the Lakme institute which is located in Hindustan levers that is the best place to learn geotechnology engineering.

What is the requirement for the nail polish? Assure they may stick to the nails for a long duration without losing the shining. So, this again mineralogy comes in the picture the bond between the material which you are using and the skin, what is the third property? There should not be toxic hazardous and there should not be any leaching into a blood vessel so; that means, they should not be further diffusion in your blood streams of the system; the same is true with the lipsticks which you guys use alright. So, the implications are tremendous. My earlier batches I use to take them to Hindustan levers Lakme center where they got exposed to design of all these things including Dove and what was the other soap which they use glycerin that brownish colour Pears yes; so, how to make a soap which is transparent, but still it act as a detergent.

So, that is the monopoly of the Unilever of UK. No one has mastered it yet, like the logic another good example I will give you in the soap industry, suppose you buy a soap today and then you keep it on the shelf in your bathroom, after two days the entire perfume evaporates. Would you use the soap or not third day onwards what will happened to the soap industry? It will close on the fourth day so; that means, all these problems require sequential degassing of the you know chemicals which are being input in the matrix at a very slow rate. Another good example is earlier days when you use to use Vim bar it used to lose its physical integrity because it used to be hygroscopic.

But nowadays you see the technology has changed and this Vim bar is still remains maintained for its life. The third problem is they should not be chemical, they should be diffusing out of the salt out of the soap matrix because of temperature, difference because of the humidity; normally humidity is going to be very high in your bathrooms and all. So, designing a soap also is a very big challenge it is not a easy task. So, this is where the magnesium oxide should not leach out of the soaps.

If magnesium oxide leaches out what does magnesium oxide does in bricks how do you make bricks? It is a binder, magnesium oxide happens to be a binder, if you do not put magnesium oxide it acts like a binding agent at elevated temperature. So, the physically strength of the soap will come because of the magnesium oxide which is present. So, if magnesium oxide itself loses out. So, will crumble; so, this type of studies become very important.

Whereas you can find some applications of these studies; fabrics particularly the rugs which normally we use the furs coats and so on; is it not? So, the more airs sorbing capacity the more heating and more conducive to wearing and body. So, these are the applications an engineering where these simple techniques can be utilized to determine their activity.

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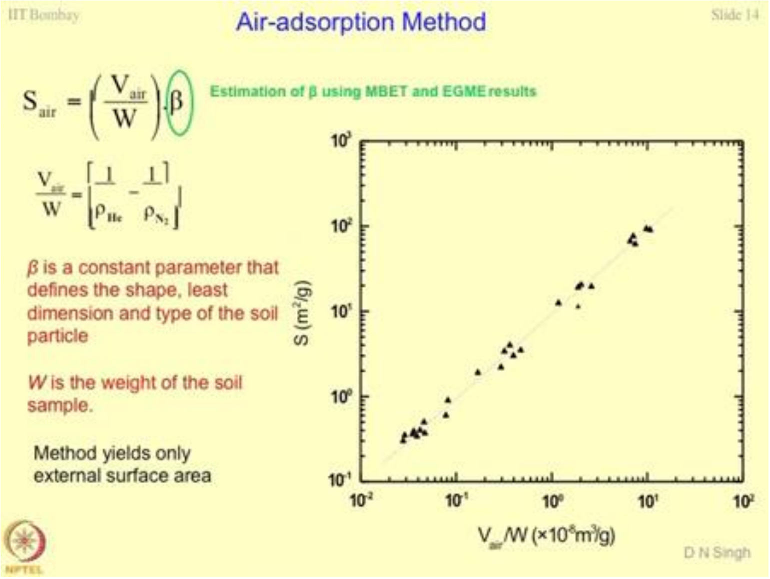

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Air-adsorption Method

$$S_{air} = \left(\frac{V_{air}}{W}\right).\beta$$

Estimation of β using MBET and EGME results

$$\frac{V_{air}}{W} = \left[\frac{1}{\rho_{He}} - \frac{1}{\rho_{N_2}}\right]$$

β is a constant parameter that defines the shape, least dimension and type of the soil particle

W is the weight of the soil sample.

Method yields only external surface area

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So, this is simple equation where you find out the specific surface here a surface area by using air adsorption method which is equal to volume of air adsorbed on the system divided by the weight of the material multiplied by some constant beta. Now, V_{air}/W is nothing, but $1/\rho_{He}$ that is the density measured using helium gas and density measured using nitrogen gas. So, beta basically corresponds to the shape of the particle, this is what we have been discussing yesterday, that you want to do the tomography of the particle you know shape and dimension and the type of the soil particle you can characterized by getting the beta value.

So, for different clays it will be different for sands it will be different combination this will be different so on. So, this surface area will be external surface area how to obtain beta value this is a big question. For determining beta, you have to conduct either MBET or EGME test so, that you can train a relationship like this. So, this is the technique where you have to adopt two methods, the air adsorption as well as EGME. So, EGME will give you a relationship like this, where a specific surface area is a function of volume of air divided by weight and the slope of this line is nothing, but beta. So, you can obtain beta from here, once you obtain the system you conduct this test again get V_{air}/W multiply these two and you get a specific surface area by using air adsorption method.

So, depending upon the requirement and the precision which you are looking for, you have to keep on changing the methodology adopting the environmental conditions and so on.

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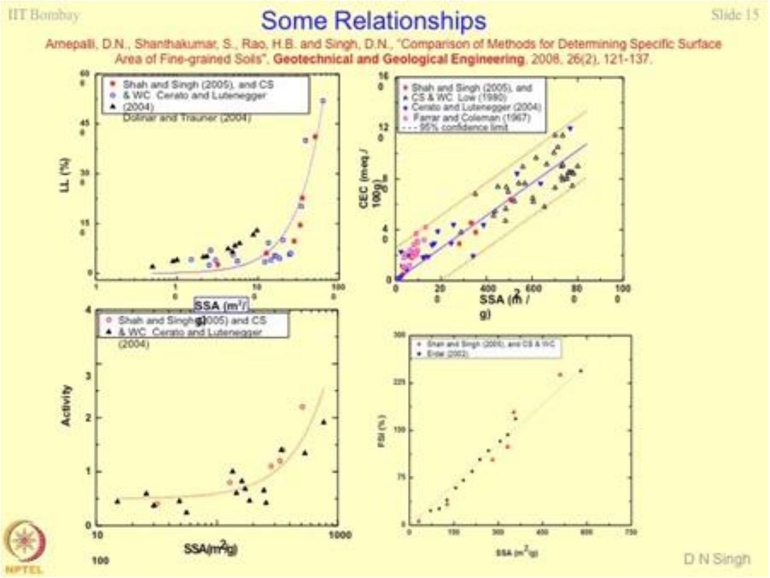

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Some Relationships

Arnepalli, D.N., Shanthakumar, S., Rao, H.B. and Singh, D.N., "Comparison of Methods for Determining Specific Surface Area of Fine-grained Soils", **Geotechnical and Geological Engineering**, 2008, 26(2), 121-137.

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Now, subsequently we develop some relationship particularly this credit goes to my PhD scholar Naidu. Dr. Naidu in fact, who did lot of work in this area determining the specific surface area of different type of soils by using all these methods and this paper was published in geotechnical and geological engineering in 2008, you can refer it if you want to go through this processes and complete methodology.

What I have presented here is only a gist of the methods but not the critical commentary particularly whenever you want to use this type of information, you should go through the literature which is available on the methodologies and their limitations. I hope you will appreciate this point that this was the effort where specific surface area has been directly linked with liquid limit. Are you satisfied with the type of test you do to determine liquid limit of the soil or you find some difficulty?

See the way liquid limit is obtained is a very you may get it on your scissors; because two persons doing the same test may end up doing different values. So, this is where we realize that SSA should be utilized to determine the liquid limit of the soils and it works out well. What you notice is as a specific surface area increases liquid limit increases you agree with these trends?

So, this side you must be having sands for which liquid limit is very less and this must be active minerals, where the specific surface area is extremely high. The basic philosophy is that it ranges between 0 this is asymptote and another asymptote come on the y axis where the liquid limit could be approximately 800%. Now, let me ask you a question, what would be the liquid limit for a typical bentonite? Have you ever tested bentonite sample yourself? Never; anybody what is your guess about the liquid limit of bentonite any number which comes to your mind Sangeetha, more than 500. While we have not come across 500, but here we are very close to 480 something the one which we have tested you are right. So, it varies from 180 to 200 to almost 500.

So, this is where we got one response, I thought you would observe this value and you can tell. So, this is somewhere very close to 500 we did one test. So, that is why they are suppose to very active materials and incidentally these are the bentonites which are calcium bentonites, not the sodium bentonites. So, this is one utility of doing this type of experiments, you can straight away check the liquid limit by obtaining specific surface area. You must have come across some literature where they talk about influence of soil contaminant interaction on liquid limit of the soil and plasticity index of the soil. Now, these issues can also be answered very easily by using this graph.

So, if I am finding out SSA, where the soil is interacting with contaminant its liquid limit is going to be different. So; that means, SSA will include this fundamental property also. The second relationship is the between the cation exchange capacity and the specific surface area and here you will find that there is a good correlation between the specific surface area and cation exchange capacity. So, more this specific surface area fundamentally though it is a physical property the activity of the clay is also going to be high; that means, in simple words cations have more parking lots available is it not on the surface of the grains.

So, more surface area more cation exchange capacity this is the relationship between activity of the soil and a specific surface area where you can see that if specific surface area is more the activity is more. Again, my idea of you know exposing these relationships to you is that you should have you should realize the point that SSA should include you know physico-chemico minerogical properties when soil comes in contact with contaminants.

So, this activity is going to be a bit different than the activity which were studied in classical geomechanics, where you talked about only plasticity index and percentage clay fraction. So, truly speaking this activity should be replaced by the word reactivity, but we have to prove this right or wrong. So, this will need some more time. So, still we are using these as the activity what and we are saying that activity is the direct function of a specific surface area. This was an attempt to check whether free soil index can be correlated with SSA. So, free soil index is a phenomenon where soil swells because of its swelling property of mineralogical interaction.

So, SSA also gets correlated quite well with free soil index of the soil. So, you can get liquid limit activity, cation exchange capacity, free swell index of the soil if you know SSA or vice versa. Now, the question is why you should rely so, much on SSA and I want to get an answer from you. See any curious mind will say why should we depend so, much on SSA; can you give me answer to this question?

You are very close to the answer, but not the precise answer, yes please any other attempt? How many techniques you use to determine LL? Only 1; how many techniques are available to determine SSA? Just count and tell me. 2 plus 4 plus 3 at least 9 10 techniques and that to these are the methodologies which are you know very precisely instrumented. So, this is where the school of thought is that if we know SSA which can be obtain very easily in 21st century most of the testing in geotechnical engineering need not to be executed alright. So, this is the moral of the story.

A day should come when the characterization of the material should be done based on only one parameter, either SSA or cation exchange capacity and you need not to do all these cumbersome tests which have being followed in the laboratories since last 30-40 years. Let me show you some more information on the application of CEC and SSA. On x axis I have plotted here W_h now W_h corresponds to hydroscopic moisture content. To your knowledge is there any code which is available to determine hydroscopic moisture content of the soils? But, did you wonder that how to determine hydroscopic moisture content of the soils.

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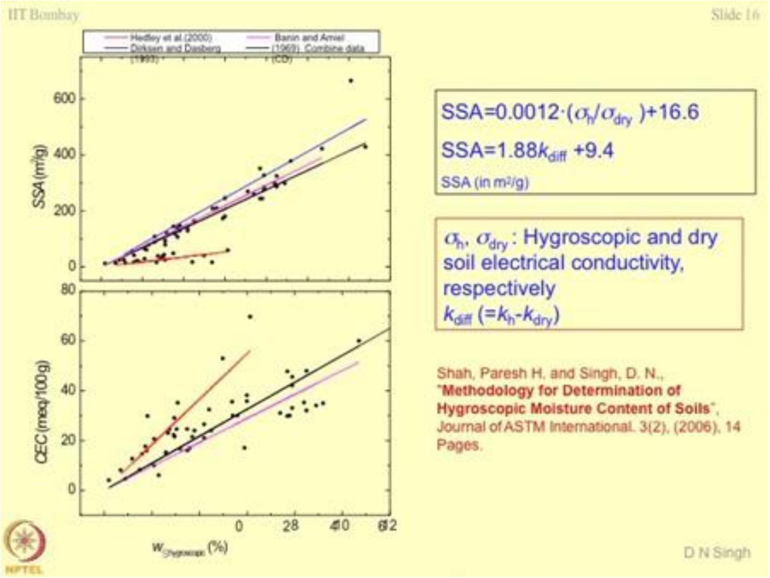

IIT Bombay

Slide 16

$SSA=0.0012\cdot(\sigma_h/\sigma_{dry})+16.6$

$SSA=1.88k_{diff}+9.4$

SSA (in m²/g)

σ_h, σ_{dry}: Hygroscopic and dry soil electrical conductivity, respectively

k_{diff} $(=k_h-k_{dry})$

Shah, Paresh H. and Singh, D. N., **"Methodology for Determination of Hygroscopic Moisture Content of Soils"**, Journal of ASTM International. 3(2), (2006), 14 Pages.

D N Singh

Well so, this was the question when one of my PhD scholars Dr. Paresh Shah will working on his thesis and this is what was published in ASTM, we came up with some equations where specific surface area is linked to hygroscopic moisture content.

Hygroscopic moisture content also is a phenomenon where the moisture present in atmosphere simply gets associated with the grains of the soil. So, you remember one of the methods of adsorption was either gas or vapor. So, it is a natural vapor which is present in the atmosphere getting adhere to the soil sample. So, this logic fits very well and what we notice is that the relationship between SSA and hydroscopic moisture content it is almost linear. So, more hydroscopic moisture content shows more a specific surface area. So, somewhere on this side we will be having sands and, on this side, we will be having active minerals. So, at least what we have done is we have mapped two extremes of the soils, that is the minerals and the sands based on this type of a relationship.

Similarly, if you plot cation exchange capacity for which the units are milli equivalents for 100 grams with respect to W_h, more W_h means more cation exchange capacity; clear? So, CEC versus SSA we have already seen in the previous class. So, these are the relationship which we get when you apply electrical mineralogy field on the soil sample. This will be discussing slightly later and we talk about electrical characterization of the soils or just to be when idea I thought this quite appropriate to put this equation here where σ_h corresponds to the conductivity electrical conductivity of the soil corresponding to hygroscopic moisture and σ_{dry} corresponds to conductivity of the dry soil.

So, this ratio is also directly proportional to a specific surface area and you must be seeing here k_{diff}, this k_{diff} is nothing, but the dielectric property of this soil. So, difference in the dielectric coefficient or dielectric constant of the hygroscopic soil and the dry soil will give you k_{diff} and this will also give you SSA. So, this is where most of the instrumentation is going on in present day you know electronics and instrumentation, where people want to determine the in-situ moisture content just by inserting a probe into it. So, what that probe is doing? That probe is simply finding out the difference between the dielectric constants and the conductivity of the selling of the material to get the surface area or cation exchange capacity and so on.

This work was published in ASTM international in 2006 methodology for determination of hygroscopic moisture content of the soil mass. From this is where actually we got this concept of drying and wetting of the soil. So, you should appreciate a point when we talk about hygroscopic moisture content this is a wetting cycle; clear? But if I want to take out the moisture from this it becomes a drying cycle for which we may use these methods for degassing the soil sample and hence making it a dry material ok. I will stop here today, if you have any doubts, we can utilize the time, yes please.

Student: Which method give correct value of SSA of all these methods which value we should take for our experimental analysis.

Normally EGME gives you the best results. So, in this paper we have shown that EGME give the best possible results out of all the methods and what is the reason if you read that paper you can follow better.

Student: Suitability of each method depends on likes a which method must be adopted now for this situation is depends on actually.

No, it will not depend on the type of the material, because after working on different type of material we realize that EGME gives you the results which are very close to the result which you get from sophisticated methods, like using pycnometer and so on. But the problem is that every laboratory and everyone cannot afford such instruments.

So, this is where we find that EGME gives to the reasonably good results as compare to the other methods, particularly BET and single point or multipoint BETs.

Any other question?

The quickest method would be gas adsorption and this gives you reasonably good results, but only thing is it will give you only surface area which is external, but if you want to get the total surface area then you have to go for EGME.

See most of the processes where chemisorption becomes very important, particularly the industry related with catalyst you know you cannot do away with EGME method there is no way, because this is where you have to ascertain the complete surface area which is exposed for interaction. So, you have to perform EGME test;

Sorry; no, we have done for rock powders also. So, this is very all type of geo materials yes, but the question is that question of the rock itself is the very abstract term; is it not?

So, which fractionally you are going to consider? But the same is valid for soils also in soils also we have different ranges of the particles. So, this is where the genesis of the idea is to fractionize the material and then find out its activity. You sieve the material go for its components and then which component is most reactive, some people are required to answer these questions.