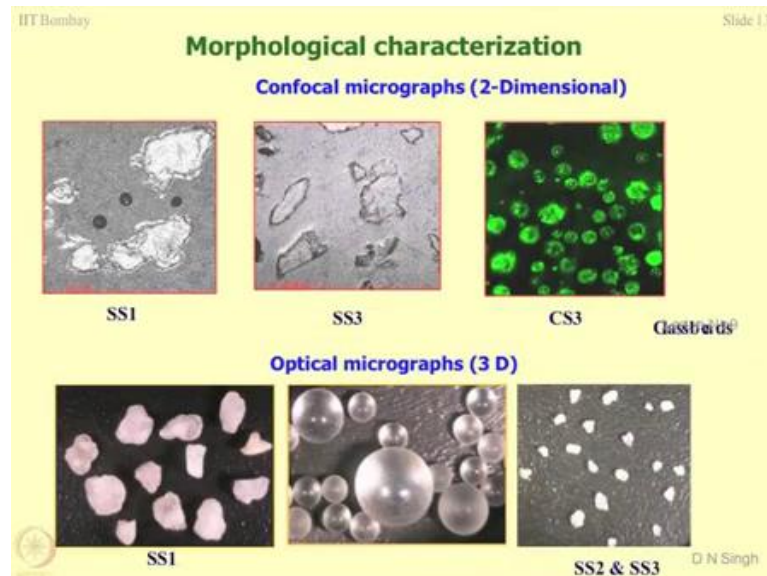


Environmental Geotechnics
Prof. D. N Singh
Department of Civil Engineering
Indian Institute of Technology, Bombay

Lecture -22
Geomaterial characterization – 2 (Morphological and physical characterization)

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Another type of characterization which is normally conducted is Morphological characterization. Morphology is basically the shape of the particle, size of the particles and so on. And this type of study was done by my PhD scholar Dr. Prasad Bartake very extensive study he has done. And I would like to show you the outcome of the study. And you should understand that what type of intricate studies are being done by people in geotechnical engineering. Now, can you identify what this system is? Here I have written confocal micrographs 2-D.

So, this imaging technique is known as confocal micrography, which always gives you two-dimensional photograph of a grain. So, these are the grains which you normally consider them as standard sand and you say the particles are almost perfect spheres. So, truly speaking the standard sands are of this size and the shape, clear? And if you look at them at three-dimensional which is known as optical micrographs, you get the real picture of the sand. So, this is how irregular shape grain particle are particle of the sands

are which normally we use in our studies when we say we are using standard sand and we assume their size and shapes to be perfect spheres.

So, here I have shown you the micrographs of sands standard sands, then cenospheres. If you remember cenospheres are nothing but the hollow spheres which are present in the fly ash. And these are the glass beads perfect glass spheres of different sizes, different diameters.

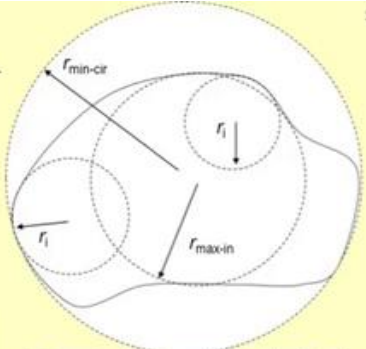
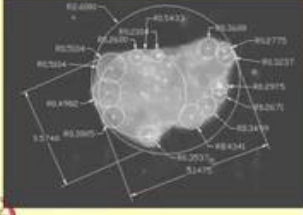
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Sphericity, S , $S = \frac{r_{\max-in}}{r_{\min-cir}}$

Roundness, R , $R = \frac{\sum_{i=1}^N r_i / N}{r_{\max-in}}$

Regularity, ξ , $\xi = (R+S)/2$

Sample	S	R	ξ
SS1	0.82	0.61	0.715
SS2	0.76	0.60	0.675
SS3	0.75	0.49	0.625
CS1	0.89	1.0	0.94
CS2	0.92	1.0	0.96
CS3	0.90	1.0	0.95

N Singh

So, when we are doing micro analysis of the systems in terms of morphological characterization, what we want is we want few numbers, so that we can classify the granular material. Unfortunately, granular material has not got much due you know from the researchers everybody talks about the fine-grained soils much more, because fine grained soils are supposed to much more reactive. So, except for crushing strength, you will not find much fundamental work going on in case of granular soils, but here we defined a granular material by using its three components that is sphericity (S), roundness (R) and regularity (ξ).

Now, this is the grain which is highly irregular all right. And then this was the model which was proposed by Dr. Bartake. If you draw two circles, one which is subscribing this, and another one which inscribing it. So, the radius of the circle which is inscribing is $r_{\max-in}$; and the circle which is outside is $r_{\min-cir}$ all right. The ratio of these to this will be sphericity (S). So, if these two radii are same sphericity is 1. For flakey particle what

will happen, $r_{\max-in}$ will be 0, so sphericity (S) number will be 0. So, this is how you can do morphological examination of clay particles.

Then comes roundness. You take a particle of the sand the way it has been shown here, and try to fit in as many as circles possible inside clear, like this is one circle, another circle, another circle, another circle and so on. So, depending upon how many circles you can fit in inside this area, you count them, find out their radius, divide by the number of particles which you have taken and then the radius of maximum radius which is inscribing the material. So, this number is roundness number.

And regularity is nothing but the average of the two. So, this is the way you can characterize the granular materials. This was the interesting scheme which we use for defining the sphericity (S) of different soils. So, these are standard sands. Which one will be the coarsest possible sand and the finest sand? This is the coarsest material; this is the finest material. So, this is how we define the index associated with this material all right. So, what essentially it requires is that you have to study at least 500, 600 grains of the sand, and come up with some average value of S and R. So, the table which you are seeing here is based on the results of about 700 grains of the sands, each sand, this was the part of his thesis.

Student: What is the use of this?

Yes, the use of this is that we want to understand how interlocking will take place, how crushing will take place, how shear strength will get mobilized in the coarse grain materials and so on. If you go by the interlocking theory, if you concentrate on this figure for some time, if another particle is sitting let's say close to this all right and there is protrusion like this. If you apply external force, what is going to happen this particle may get chopped from this plane and that would be one of the ways of defining the crushing strength.

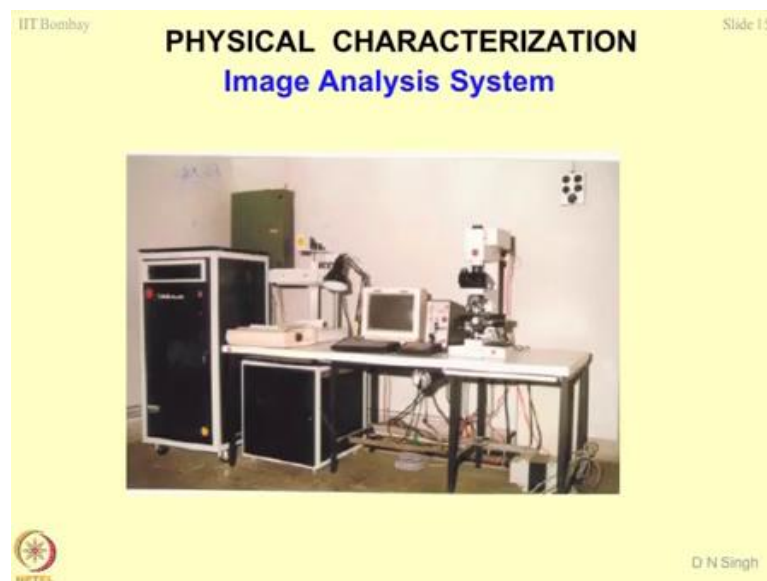
So, crushing also has different manifestations associated with it. It is only the breaking of one teeth of the grain and it may give you a picture which is not correct. So, these types of studies are required to understand at granule level what is happening in the material. What comes to your mind, why this type of study should be done? Any thought which comes to your mind?

(Refer Time: 07:03)

Any other question related to this or may be answer or idea what can be done further with this? Suppose, if I give you this map, what else you can do? See this is genesis of very big research idea. Most of the time you talk about void ratios you know e_{max} e_{min} which are very gross in nature, where you assume lot of things, cubic arrangement, rhombic arrangement of the coarse grain materials based on perfect spheres. But what we are trying to show here is that those models are not correct, because you have not looked into the micro structure of the grain all right.

And the bottom line is that we have supposed to do something new. So, for the heck of doing it, we have to do all this. Unless you propose new ideas your papers and your ideas will not sell. So, if we have to survive in our profession, you have to think of new things and we have to keep on publishing, it has to be some new concept. So, this is what is known as reading between the lines.

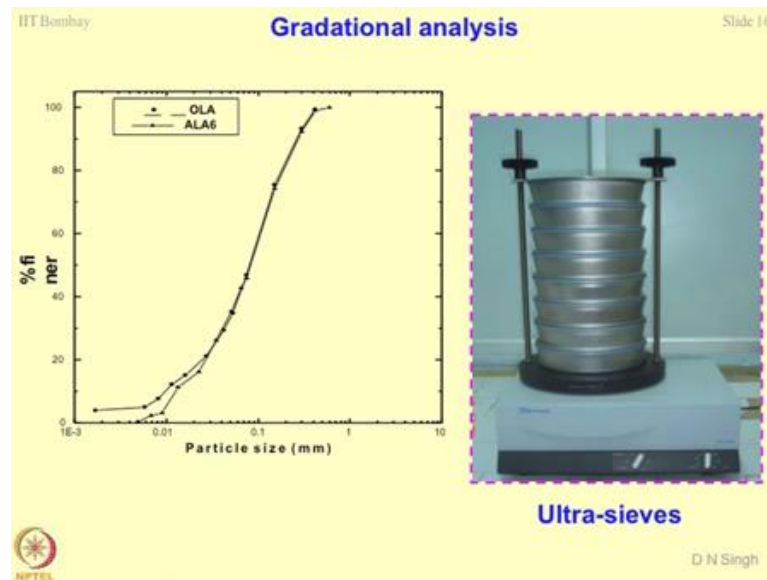
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So, let us talk about physical characterization. Which most of you have been doing quite extensively. For particle size analysis, you normally do sieves or hydro meter test. Well this is a sophisticated instrument which is known as image analysis system or particle analyzers also sometimes call it laser, laser particle analyzer also sometimes people call it. So, there is a microscope kept here - table top. You can spread your sample on this

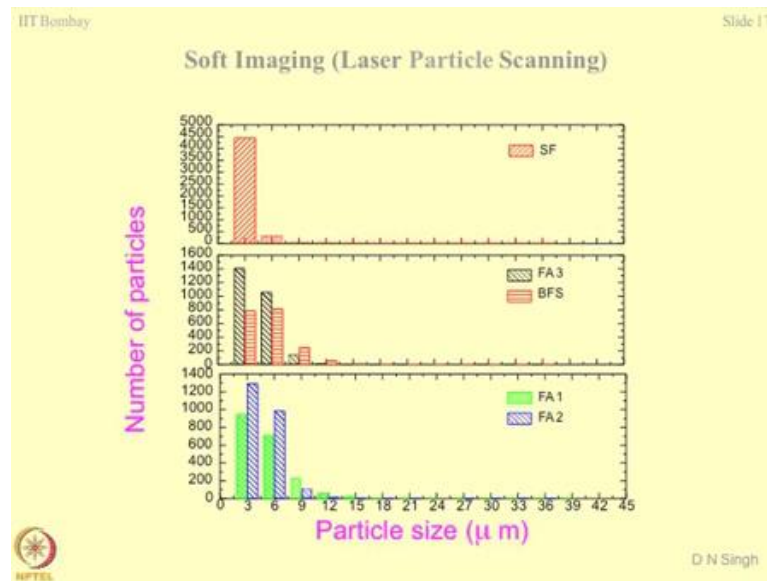
pedestal. And you can freeze the picture. There is a PC connected to this on which you can see on the screen you can see different types of shapes of the particle. And basically, it is statistical analysis, I will show you the result of this.

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Sometimes you can use ultra sieves, you might have seen in the lab where you can do dry sieving to get the particle sizes. And what you end up is with a particle size distribution graph. So, in my opinion, whenever you do a direct shear test on sands, you should always work out its particle size distribution before shearing and after shearing and you should see the difference. Why it is required? The difference should be studied to ascertain that no significant change has taken place in the material which has been sheared clear. So, these are all parameter which are normally considered when you are talking about the mechanical response of sands.

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Well, these are the results of laser particle scanning system or the soft imaging system which I have shown you earlier. We have to literally count the number of particles which are present in a batch and the particle size. So, what this analyzer does is for different particle sizes it becomes it gives a range. So, let us say from 2- 3μ the number of particles is approximately 900. So, literally have to count one by one, 900 particle, 1000 particle and so on. Research is not so easy and each test you have to repeat at least 5, 6 times. So, that you may get the representative values this was done by Dr. Krishnayya who was at JNTU. This is from his PhD thesis.

You will notice here that if you do the analysis for silica fume, first of all the question comes that why should we do this analysis for silica fumes. Cannot you find out particle size distribution by some other method? You know? Why? First of all, they all are sub micronic particles and they are much lighter than air. So, even you make a slurry of it, nothing will settle down. So, hydrometer cannot be done. Why sieving cannot be done?

Student: (Refer Time: 12:00).

Yes, there is lot of losses and nothing will pass through the sieves. So, in this such situation, soft image analysis becomes the best option where you can magnify the grains and you can see their shapes and you can count what is the size.

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Property	FA 1	FA 2	FA 3	GGBFS	SF
G	2.03	2.3	2.38	2.84	2.1
Specific surface area (cm ² /g)	2988	3602	5048	4073	200000
Sand size (>4.75 mm) %	0	0	0	0	**
Silt size (0.002-0.075 mm) %	100	95	90	97	**
Clay size (<0.002 mm) %	0	5	10	3	**

** Not applicable

Particle size Range (µm)	Number of particles					Percentage of particles				
	FA-1	FA-2	FA-3	GGBFS	SF	FA-1	FA-2	FA-3	GGBFS	SF
0.0-3.0	944	1295	1410	789	4451	47.6	53.0	53.1	40.3	90.6
3.0-6.0	709	989	1061	816	343	35.8	40.5	39.9	41.7	7.0
6.0-9.0	228	116	148	247	48	11.5	4.8	5.6	12.6	1.0
9.0-12.0	57	17	21	59	33	2.9	0.7	0.8	3.0	0.7
12.0-15.0	24	11	4	16	17	1.2	0.5	0.2	0.8	0.3
15.0-18.0	6	2	2	9	6	0.3	0.1	0.1	0.5	0.1
18.0-21.0	4	1	1	5	2	0.2	0	0	0.3	0.0
21.0-24.0	2	0	0	5	3	0.1	0	0	0.3	0.1
24.0-27.0	1	1	0	2	3	0.1	0	0	0.1	0.1
27.0-30.0	2	1	1	5	2	0.1	0	0	0.3	0.0
30.0-33.0	2	6	3	3	4	0.1	0.2	0.1	0.2	0.1
33.0-36.0	2	1	2	3	2	0.1	0	0.1	0.2	0
36.0-39.0	1	0	0	0	0	0.1	0	0	0	0

D N Singh

This is the table which gives you different properties of the materials. So, fly ash as you can have specific gravities of 2.0, 2.3, 2.1 for silica fume. A specific surface area if you look at it is almost 200000cm²/g that means, 20m²/g is a specific surface area of this material. So, the question is how to find out the specific surface area of this type of a system. I will show you how specific surface area are determined.

And this is the analysis for different materials fly ashes and blast furnace slag, silica fume. What you notice is in the range of 0-3µm, you have approximately 4,000 particles. So, your hydro meter analysis cannot be done for particles less than 2µm. So, 0- 3µm range basically corresponds to 0.003µm range, but then those who are working in concrete and HVFC, there they require this type of classification of the material.

So, you will find that in the range of 36, 39µm nothing is available for this material and that is the reasons why fly ashes and silica fume and GGBFS they are used in concrete. So, if you remember the basic idea of using these materials is that you can seal the pores and you can make a concrete which is much more durable. Is this part clear, any doubts? So, from the number of particles, then you can compute the percentage of particles and then you can plot it in a form of a histogram the way it was done earlier.

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The second attribute of the material is specific surface area. Why specific surface area is so important to be determined?

Student: It shows the reactivity of the material.

It shows the reactivity.

Student: Of a.

Potential, all right. How much reactive a material would be? So, finer a material more surface area highly reactive with the water, environment, contaminant and so on. So, that is why most of the studies in present day geomechanics deal with precise determination of specific surface area. Now what is the difference between surface area and specific surface area? Why do you call it specific?

Student: (Refer Time: 15:05).

Correct. So, specific surface area is always surface area divided by grams per unit gram of the material, so that means, 1 gram of the material of silica fume gives you specific surface area 20-meter square. What is the size of hockey field, any guess? You are interested in any sports? What is the size of swimming pool, Sneha?

Student: (Refer Time: 15:36) 50 25, 50×25 .

No, the new one is 50×25 . The old one?

Student: $16.5, 25 \times 16.5$.

No, it is 28×23 , 22 approximately meters all right. So, this comes out to be approximately how much 400m^2 clear. So, $1/10^{\text{th}}$ of that will be approximately 40m^2 , so that is the surface area of each particle of silica fume, just imagine. So, when you go for assembly of the particle the reactivity is going to be so high is just give you idea about what is surface area is all about. See this is why mineralogy which has been ignored till now by geo technical people is becoming more and more is coming more and more lime light and everybody wants to study at the micro level and this is where all this characterization schemes and tools become very important. I hope now this point is clear.

So, when we talk about specific surface area, there are different techniques. These instruments are very expensive all institutes and organizations may not have them that is why Government of India had set up advance centre for research in different corners of the country. They were five you know CSRE type in the entire county these are known as Regional Sophisticated Instrumentation Centres, one of them is located IIT, Bombay which is known as SAIF.

So, the first instrument which is normally used is BET nitrogen adsorption. This is now available in chemical engineering, metallurgy and chemistry department. To my knowledge the recent version of this instrument costs about 50-60 lakhs. Then second instrument, not sorry it is not an instrument method absorption of ethylene glycol monoethyle ether. This work was done by Dr. Naidu; he is an expert in determining surface area of the soils, now he is assistant professor at IIT, Chennai.

So, he has spent enough time in coming up with a methodology and showing that which method out of all these is the best method for determining specific surface area of soils. Then there is a method known as methylene blue - MB dye method there is a dye which you have to put in the soil mass depending upon the sorbing capacity of the soil, the concentration of dye gets reduced. And then you can use spectrophotometer to determine how much concentration of dye has been absorbed by the soil mass deduct the two find out the percentage and that is what is related to the surface area.

Mercury intrusion porosimetry which we have shown you the other day when you came to the lab. MIP is also used for finding out the surface area of the grains, but not a very good technique I would say. Helium gas pycnometer can also be used for determining the surface area and Blaine's apparatus. You must have heard I am sure that you might have used this Blaine's apparatus in your under graduate for determining surface area of cement or sands.

Student: (Refer Time: 19:07).

Student: (Refer Time: 19:10).

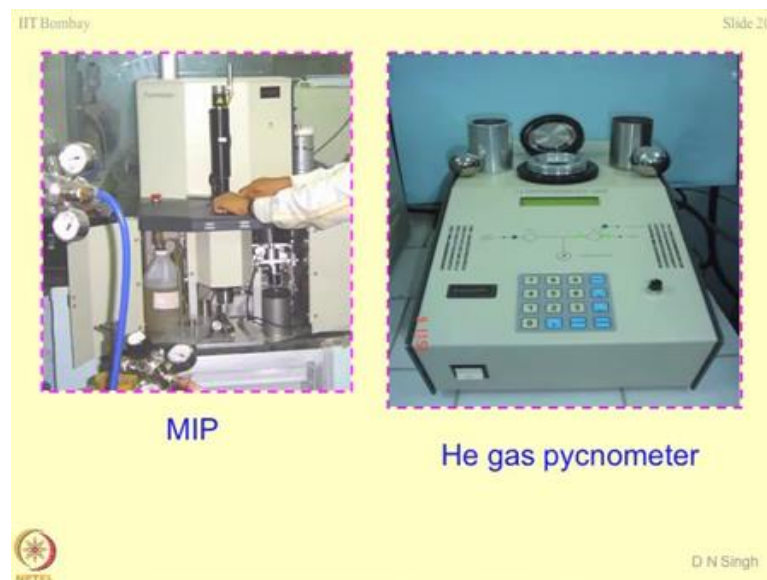
ok

This also known as air permittivity method.

Student: Blaine permeability

Gain permeability.

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So, this is the MIP - Mercury Intrusion Porosimetry, you have already seen that helium gas pycnometer you have seen.

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Blaine's Air Permeability Apparatus


(ASTM C 204)

Portland cement as a standard reference material

Specific-surface area (S_B)

$$S_B = \frac{S_s(1-e_s)\sqrt{e^3}\sqrt{T}}{\sqrt{e_s^3}\sqrt{T_s(1-e)}}$$

S_s is the SSA of cement (= 0.346 m²/g)
 e is the void ratio of the sample
 e_s is the void ratio of cement (= 0.5)
 T_s is the time of manometer drop for cement (= 77.18 s)
 T is the time of manometer drop for the sample



D N Singh

Now, this is the Blaine's permeability apparatus for finding out the specific surface area. Now, this is as per ASTM C 204 for admixtures and pozzolanic materials. And issue is that you use here as reference material the Portland cement. And for Portland cement the specific surface area is 3460 cm²/g. So, what do you think, that this method can be applied to any material or there are some constraints limitations?

Student: So, we cannot go below the reference.

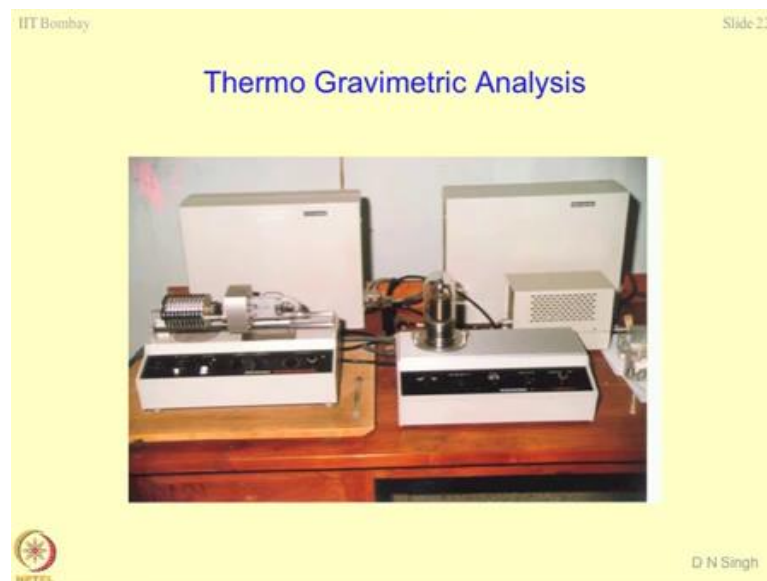
Yes, not below, not below, higher. So, the materials which are finer than cement, particularly silica fume where the surface area is of order of 20, 40 m²/g, most of the synthetic material zeolites resins in chemical application whatever they are using you cannot use this technique. So, if you would use air permittivity method, you will end up in getting wrong results. So, as per this method, the specific surface area can be obtained by using this equation where SSA is the surface area of cement which is standard 3460 cm²/g; e is the void ratio. There is a tube, and above the tube there is collar in which you keep the sample.

So, it is sort of manometric tube where you can pump the air in and you can release this air, so that the air passes through the sample. So, how easily air passes through the sample this is what you are trying to judge over here. So, e is the void ratio of the sample in this tube; e_s is the void ratio of the cement. These tests always done in duplicate one

with the material for which you are trying to find out the surface area and second test would be done on cement. So, with respect to cement, you can always work out the surface area. And T_s corresponds to the time taken by the manometric drop for the standard material that is the cement and T is the time drop for this sample. So, using this equation you can work out specific surface area.

Not void ratio, e_s this is a typical example. So, the container which is shown here when you pack the cement in this container, it will give you typical value of 0.5 of e_s normally. It is an old method. Now, I should say that people do not use this method and they normally go for the method which I shown you earlier. Just to give you an idea that where the research is in you know cement in cement manufacturing, people are trying to min maximize the surface area of the cement. So, if you can create a cement of the order of let us say $50 \text{ m}^2/\text{g}$, that would be wonderful.

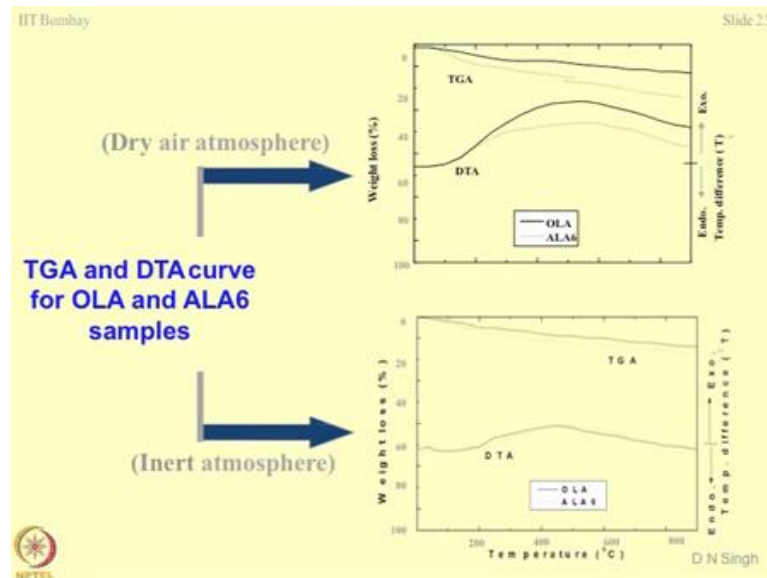
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Well, this instrument shows you a TGA analysis - Thermo Gravimetric Analysis. Why do you think that this type of studies is required? You have been finding out gravimetric moisture content all the time is it not by keeping the wet soil in oven. And after it dries up you take it out and find out the difference in the weight, and then you can work out the moisture content. Thermo gravimetric analysis normally done to see the stability of the material when it is heated up. So, you should not use a material which is thermally instable, particularly let us say when you talking about admixtures or the materials for

their application in concrete. At elevated temperature level what will happen, they will simply disintegrate. And if they disintegrate, the structures are going to collapse.

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Now, this is a typical result which I am showing you for a TGA and DTA analysis. TGA corresponds to thermogravimetric analysis and DTA corresponds to differential thermal analysis. These are two methods of finding out the thermal stability of the system. So, in thermogravimetric analysis what to do is, you heat the sample for at different temperatures clear.

So, with respect to temperature, you find out what is the weight loss. From y-axis, there is a weight loss, percentage weight loss. So, depending upon the material what is happening here, as the temperature increases the weight loss increases this all most 0-100% weight loss. So, as temperature increases, the weight loss increases and then it becomes constant, all right. However, a DTA shows you there is hump. Now, what this hump corresponds to? This hump basically corresponds to exothermic or endothermic reaction.

So, if this hump is above the line of this starting point, it is always a exothermic reaction, that means, when systems is being heated up it shows heat emitting out of it; vice versa if this dips down, it takes heat from the system. So, for designing different types of concretes, what type of cement should be use in what type of environment, in every

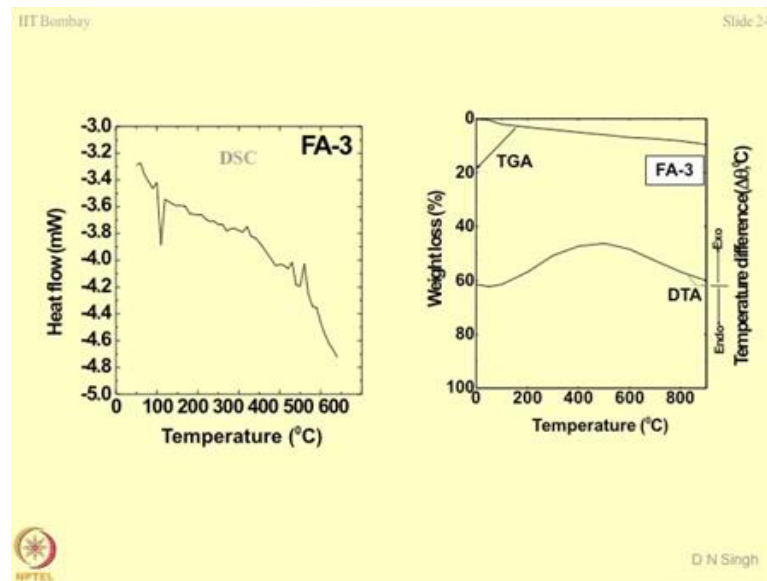
environment you cannot use the same OPC of cement. These types of studies become quite useful. And these types of studies are normally done in two atmospheres. So, the first graph which I have shown you is normally in dry air.

The same experiment is replicated in inert atmosphere like nitrogen gas. So, what is the difference between the two, in what way you can use this two information? If there are some organics which are present, they will not show up in inert atmosphere. So, the difference if you want to make out what is the organic content, what is hydro carbon content associated with the geometrical, you can do this simple test and you can work out what is the amount of organic contents which are present, what is amount of volatiles which are present in the material. So, again this is the very good scheme to differentiate the amount of contamination which is present in the geomaterial it is.

Now, if I ask you question can you differentiate between this response and this response and what is your intuitive feeling What it essentially means is that in inert atmosphere, the exothermic reaction is less as compare to correct. So, this happens to be a very active material which reacts with the air or the atmosphere. And under inert atmosphere condition also it has a tendency to show you some exothermic reaction. So, you have to deal with this material very carefully. It is a typical example of one of fly ashes which we have been working with.

So, the whole idea of showing all this is that lot of intricacies are involved in you know studying the material behavior. This is basically the difference between the activated ash and the natural ash or the non activated ash. I do not want to go in all those details, I just wanted to show you what is the application of this type of studies. Any suggestions or doubts, let's proceed further?

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This is a good relationship which I wanted to show you which is response in a DSC. DSC also stands for doctor of science, highest degree in engineering or technology or any subject much higher than Ph.D. But here DSC corresponds to any guess Differential Scanning Calorimetry. So, what happens here is you heat the sample slowly and then try to see whether the heat flux is in the sample or out of the sample, again the same thing either exothermic or endothermic.

Now, whenever there is a drop sudden drop what is the meaning of this, this shows thermal instability clear, that means, material is becoming instable at this point. What it indicates to if you are using this material for fire resistance application, it is a misfit. So, as I told you after you know this twin tower collapse, civil engineering lot of people are concentrating on now thermal fluxes, thermal heating procedures and so on in designing these structures.

So, this is a steel material, admixtures are to be tested for thermal stability. So, this indicates that the material which you are dealing with is not a very good material to resist 100°C, because there is thermal collapse or instability. Now, this is a result of a typical fly ash which I have been showing you. So, most of the fly ashes, they have calcium oxide in them and because of calcium oxide, they will show you exothermic reaction.

Now, if you are trying to use a mineral for your you know as a cosmetic, what type of minerals will use, a mineral which shows exothermic reaction or endothermic reaction? It should cool for facials particularly ladies use is it not. So, you have to have mineral when it to contact with water, it should give you cooling effect. So, all your ayurvedic treatment, they use the minerals where they take the heat from the body and they show you cooling effect all right. So, this type of mineralogy can also be studied by using this technique. Now, this project we were doing for Pidilite industry by the way for designing their brand, you know the brand which they make. What is the name of the brand Fevicol?

Student: Fevicol.

The biggest question is that why Fevicol should be studied, what are the gradients of Fevicol? Fevicol has lot of minerals of clay or illite or kaolin. For that matter, your Colgate toothpaste also has lot of kaolin, is it not? You agree or not? Now, the question is if you make a paste which cannot be squeezed out of the tube, what is going to happen? You get up in the morning you try to press it and nothing comes out.

So, this becomes very interesting problem related to rheology of minerals, where you have to contribute to the society. Similarly, the mineral which are use for you know remediating the nuclear waste, hazardous wastes and so on, the applications are tremendous you cannot believe. Any glue, which you use mostly having a binding material as a mineral. So, this is where the mixing of the mineral with the glue rheology of the paste, thermal stability of the paste, the type of you know color which you are adding to this and the type of perfumes which we are adding to this they should remain there becomes a big issue.