Environmental Geotechnics
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Lecture- 21
Geomaterial Characterization -1

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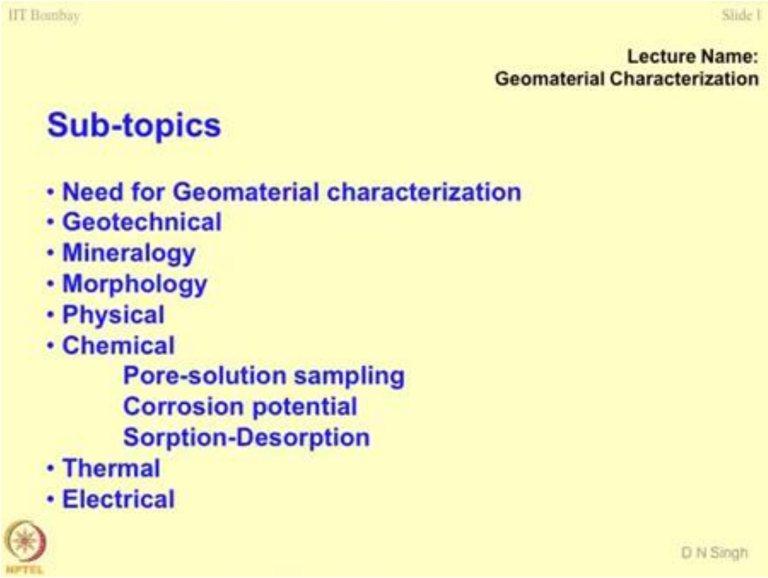

In today's lecture, I intent to talk about geomaterial characterization. And the subtopics are need for geomaterial characterization, I have emphasized upon this several times that why do we require characterizing the material particularly geomaterial, the soils and rocks or the waste form of the industrial waste. When we talk about geomaterial characterization, the first topic is geotechnical characterization, mineralogy characterization that is how to determine the mineralogy of the material. Next is morphology, particularly dealing with the shape of the grains and the size of the grains. Physical characterization which most of the time, you have already done it, you are aware of how physical characterization is done. Chemical characterization, some part of this you might have covered in your under graduate. This is where we will discuss about pore-solution sampling, how the pore solution is obtained from this soil sample.

Application of pore-solution sampling would be in determining corrosion potential of soils or the geomaterials, and this also falls in the category of chemical characterization. Followed by the sorption desorption test, which describe the reactivity of a geomaterial with environment or contaminant. Followed by thermal characterization of geomaterials and lastly the electrical characterization of the geomaterials. In today's lecture I would try to finish the physical characterization part, and then most probably in the next lecture, I will be talking about the chemical characterization.

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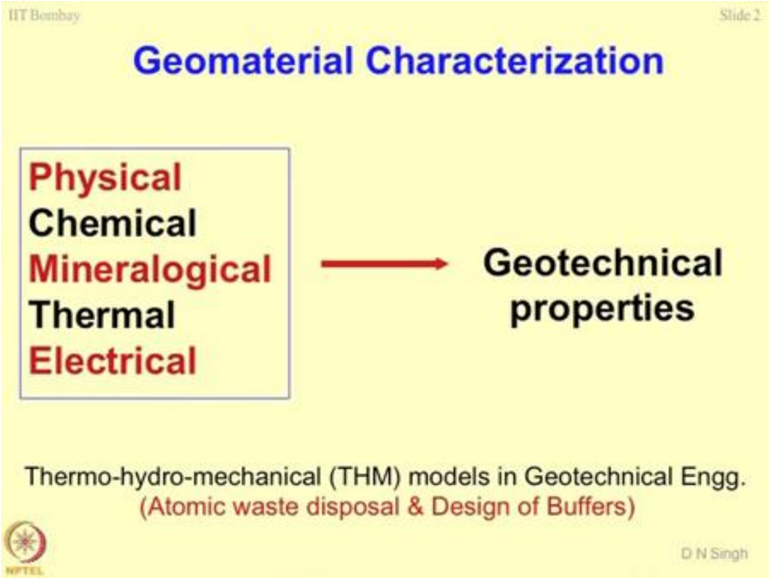

Now, what is meant by geomaterial characterization? The first letters are not coming here it seems, the physical characterization, the chemical characterization, mineralogical characterization, and thermal characterization. So, basically what we want to do is we want to study how these things influence the geotechnical properties of the material. Till now whatever we have discussed in this course, you must have understood the point that definitely all these properties are going to influence the material properties, how they are going to you know exhibit their response in the nature. So, this is where we will actually try to understand how physical, chemical, mineralogical, thermal and electrical properties are going to influence overall geotechnical engineering property.

Now, why this topic is becoming very important these days, it's becoming very important these days because of most of the times when we deal with hazards and toxic waste particularly radioactive waste, so this is where the basic question is how to design the buffers for waste. And when we say buffer these are the materials which will be acting as a retardant to the activity of the waste. Now, when this material comes in contact with the waste which is having attributes of atomic waste in terms of its radioactivity, temperature, concentration; a phenomenon which is known as thermo hydro mechanical phenomena becomes very important.

And in present scenario most of the laboratory most of the lectures are working in the area where they trying to see the response of geomaterials under thermo hydro mechanical situations; that means, how hydraulic properties and mechanical properties of geomaterial change when the material comes in contact with thermal fields. So, this is a very good research topic in present scenario. Most of research person in western world, they are emphasizing on these types of models. But you will appreciate a point that to study THM models, you should understand how material is going to behave under different energy fields which we have discussed earlier.

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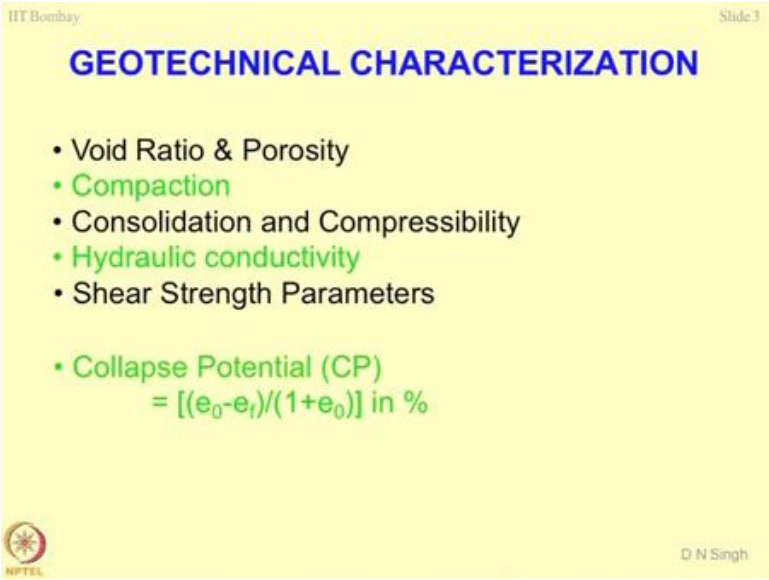

So, let us start with the geotechnical characterization. Most of you are aware of what is the significance of geotechnical characterization, what are the attributes of this type of characterization. The first one is we would like to study what is the void ratio porosity of the geomaterial, because this is the gross property which helps in understanding the response of geomaterials towards permeation of a flux that is the water or the gas or whatever, so the fundamental property of the geomaterial. Then we try to study the compaction characteristics by doing proctor compaction, modified proctor compaction and so on. Then we talk about consolidation and compressibility characteristics of the geomaterial. The next is hydraulic conductivity and shear strength parameters.

And this is followed by parameter which is known as collapse potential. I am sure that you must have some test while doing consolidation and compressibility characteristics of the material to determine collapse potential of the soil. Now, this parameter becomes very important in the analysis particularly when you are working on soil which are highly compressible and which may collapse because of inundation due to water or exposure due to water or pooling of water let us say. Normally, the collapse potential defined in terms of $\Delta e/(1+e_0)$, and this term is always in percentage.

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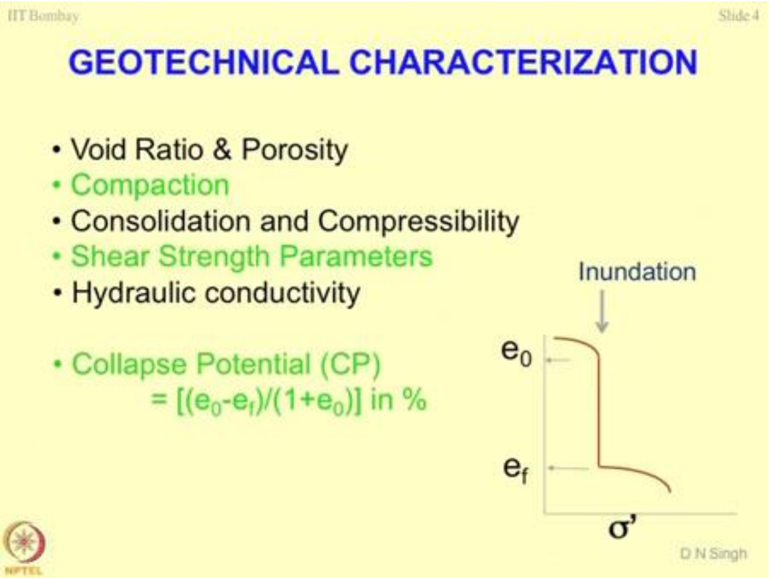

Now, to understand this phenomenon, you can consider this figure where the void ration (e) is plotted located with respect to the effective stress (σ'). Now, if you do a conventional consolidation test, your e vs σ' curve would be a non-linear curve. So, as σ' increases, e decreases. But suppose if I decide a pressure or σ' at which an alloying material to get flooded water, in that case what is going to happen? Now this is what is known as inundation that means you flood the system with water. And particularly when we talk about collapse potential, the starting point is the dry soil mass, you packet in a consolidometer ring and then apply certain normal stress. At this sustain loading, if I allow water to flow into the sample what is going to happen? There will be a sudden drop in the void ratio and this is what is known as collapse of the material.

Now, what is the practical application of determining the collapse potential? Can you can you tell me? Can you guess that what need to be best possible application of this type of philosophy? You think of a situation where the waste has been piled up near some industrial activity or near a mine. And what happened is this waste has been piled up loose form and then comes rains, clear. So, what is going to happen? A system which is packed to a loose form when it comes in contact with water or gets flooded with water, the entire heap may collapse. So, this is where these types of test can be simulated very easily in the laboratory.

Now this is one of the examples of how waste environment interaction can be studied as for as structural properties are concern, because this collapse is nothing but a structural collapse clear. So, if you know the initial void ratios, and if you know the final void ratios after inundation, you can work out what is the susceptibility of this material towards collapse. So, this parameter becomes very important when we design heaps or the landfills. Is it ok, any doubts?

Student: (Refer Time: 09:36)

Yeah, collapse potential is always defined for a given normal stress.

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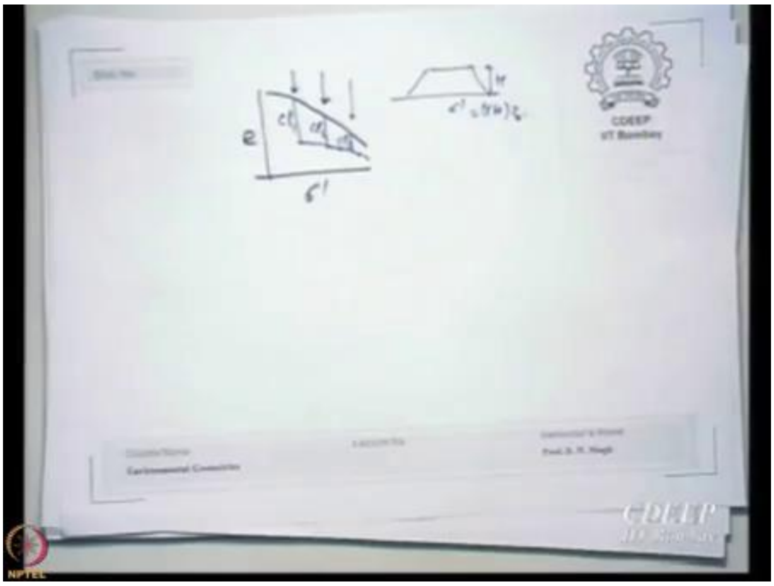

So, incidentally what will happen is that see normally if you plot e vs σ’, this is a normal consolidation graph. Now, if I want to find out for a given embankment which is of height H, I can compute the σ’ value which is nothing but γ×H and some factor. Now, if I inundate at this point and I sustain the loading, what is going to happen; this system is going to collapse like this. And then if again I continue loading the system this is what the response would be. I can do further; I can apply inundation somewhere here. Now what has happen? The system will collapse like this, it will continue like this. If I sustain this point, what happens? This is the collapse of the material and so on.

So, if you consider CP_1, CP_2, and CP_3, which are nothing but a collapse potential I think you can understand that for a higher sigma prime, the collapse potential keeps on decreasing all right. So, this is the good example of how you can design your system for sustain loading. So, this is what most of you would be dealing with when you design your landfills or the disposal systems particularly when this stacking is in dry condition. Is this ok? Any other question?

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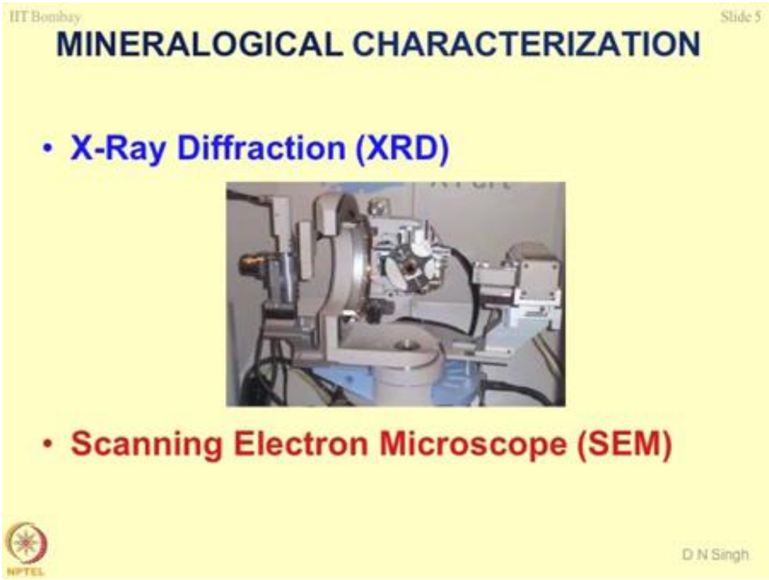

So, most of you are aware of how to characterize materials by determining its geotechnical characteristics. Now, let us study how to find out the mineralogical properties that is mineralogical characterization. The most important and useful tool or device or methodology would be to conduct XRD that is X-Ray diffraction. I have shown here a setup where XRD machine is shown. This setup is available in our institute at SAIF and in Metallurgy department. So, we take help from them to determine what is the mineralogical composition of a material which is known as XRD.

So, essentially what happens is that you have some beams of X-ray which is which are falling on the sample and then whatever diffraction is obtained can be obtained by using some principle unit. The second tool which is used for determining mineralogical characterization of the material is a scanning electron microscope which is SEM all right, where you can find out the composition mineralogical composition of the material in the best possible manner.

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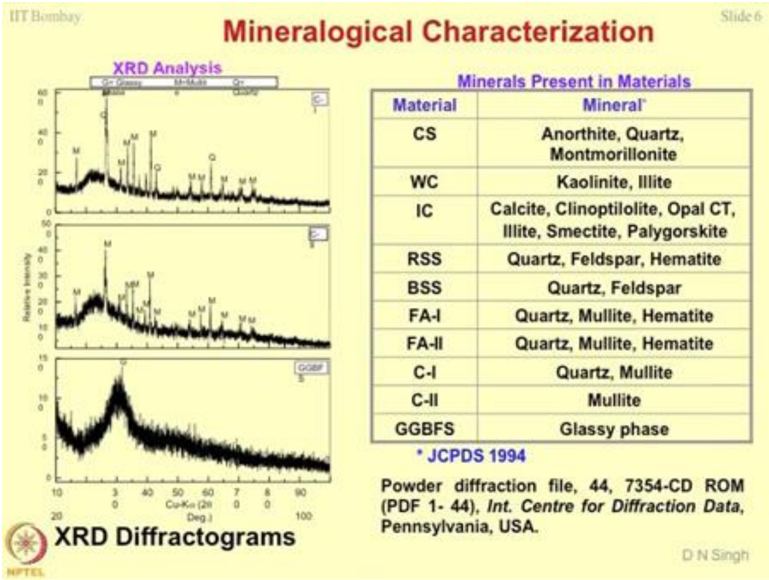

A typical result of mineralogical characterization is shown over here for different materials which I have included in my today’s presentation. These materials are a CS type of soil which is known as a silty clay, a white clay which is nothing but pure Kaolin, some chalk samples in Israeli chowk, red sandstone, brown sandstone, different fly ashes, cenospheres of different types, and GGBFS or the blast furnace slag.

Now, if you look at the XRD analysis or XRD patterns which are also known as XRD diffractograms, you will get something like this. You must be noticing a lot of peaks. Normally, XRD is conducted by using a filter in this case filter was copper k-α (alpha). So, copper k-α is the filter which is used for doing this analysis, and we use the angle 2θ, where θ is the angle of incidence with respect to normal of the X-ray beam.

So, normally this analysis or scanning of the sample is done from 10-100 degrees. And depending upon the minerals which are present, we will find different type of peaks here. So, each peak corresponds to a particular θ angle. Now, eessentially what you are doing is if you know the radiation source or the filter source, you know in the wavelength (λ) of the wave all right. And then by using Bragg’s law:

$2d \sin\theta = n\lambda$.

So, here d = the spacing between two planes. So, this way you can work out the value of d which happens to be a characteristic value for a given mineral is also known as lattice structure.

So, corresponding to a particular theta what you can ascribe is you can ascribe a particular mineral like in this case M, M corresponds to mullite, Q corresponds to quartz and so on. So, whatever minerals are present, you make it a picture of these minerals in the diffractogram. Now, the question is how are we going to read this diffractogram. For reading this diffractogram in today's world, people use JCPDS files. JCPDS files were given by the powder diffraction centre which is located in Pennsylvania USA, it's a material characterization lab. And they have come up with standards different type of standards. So, this is a CD-ROM on which all the minerals are listed with d values. As I said d are spacing between the two adjacent atoms of the material. So, this way you can identify up to fourth decimal place the value of d, and that value is corresponding to a particular mineral.

Now what else you find the difference between this figure, this figure and this figure, can you differentiate between 1, 2 and 3 figures? Yes, one is the height of the peak, clear, that is not very much important in X-ray diffraction analysis. Do you find any other difference?

Student: (Refer Time: 16:38)

Correct. So, here you will find a band. Now, this is what I have been talking about some time back that depending upon the phase mineral which is present in material, you can define its activity or reactivity. The basic difference between 1, 2 and 3 is you have here less peaks, is it not. However, in these two cases, you have lot of peaks. So, what is the significant of this? Each peak corresponds to a crystal; that means, each peak corresponds to a crystalline phase. So, a material which has more crystals, more crystalline phase is always going to be less reactive, it is a passive material. However, if peaks are less and you got a band, it shows that these material is going to be highly reactive because here alumina silicates and other minerals they are present in glass form. And glass also can be crystalline as well as amorphous. So, here the glass happens to be amorphous ways.

So, this phase of the material is highly reactive. So, the moment you put water into this or the moment interaction between water and this material takes place immediately, the gel formation takes place. So, when you are making concrete which material you will prefer out of the three.

Student: Third one.

Third one. So, incidentally this corresponds to GGBFS that is blast furnace slag. So, blast furnace slag you will see further that why it is used as the replacement material for cement. But the first threat is the mineralogical this material happens to be quite active, because most of them minerals are present in a amorphous phase, not in the crystalline phase. So, this is how you can differentiate between the materials immediately. And then once we had differentiated the material, you can use it for a specific purpose.

So, suppose if I give you different materials, and if I ask you to select materials which are the right candidate material as a backfill material, and the right material for replacement in cement. So, which type of material you will use as a backfill material and which type of material you will use as a cement replacement material out of these three?

Student: (Refer Time: 19:01)

So, first and second becomes a good material for filling or land filling back filling, raising the foundation, platforms and so on.

So, if you want to construct embankment out of the fly ash clear. So, this I hope now you it should be clear to you that how easily you can define and use the material for a specific purpose, this is for clear? I have tried to give the maximum possible information analysis wise. And based on this information immediately, you can say that this material is best suited for this purpose or not.

Yes, Suchitha asked a very good question that this is basically a qualitative way of defining the minerals which are present in the material. Now, is there any way to quantify? Can you guess is there any method to find out the quantitative value of the mineral which is present? For that what you have to do is you have to take the purest form of the mineral, do XRD, and super imposed XRD diffraction pattern on these graphs. Depending upon the relative magnitude of the peak which you are getting that is the relative intensity, you can define depending upon 100 percent peak length in the pure mineral and the height of the peak in this case you can find out what is the percentage of the mineral present. So, this is how you can quantify the amount of minerals which are present in a geomaterial as well. This was a good question.

So, this type of analysis we are using quite a lot in our study to differentiate the potential of the material for a specific purpose. Now, here I listed some minerals which are present like anorthite, quartz, montmorillonite; in white clay normally, which is nothing but lllite type of soil or kaolinite type of soil; calcite, clinoptilolite, opal CT, illite and so on in chalk samples. These are the chalk samples which we had got from Israel and they are the best possible accupers, their porosity is are approximately 38-40%. So, in our studies we are using samples collected from Israel is known as rally chalk. And then you trying to study the response of the rally chalk with respect to our own materials like a red sand stone and ground sand stone.

In typical fly ashes, you will find quartz, mullite and hematite. Hematite is a compound of iron. Then I have used some cenospheres where we have pure quartz and mullite, incidentally if you can recover these cenospheres, you can do lot of industrial applications with them particularly manufacturing good dialectic material. So, these are purest form of the quartz, which is normally used in a dialectic in your chips and electronic circuits and electronic devices. You must be noticing here that for GGBFS I have written glassy case, there is no crystal present expect for the glassy phase and that is the reason it is highly reactive. So, is this part clear that how based on simple XRD analysis, you can differentiate the material and you can at least suggest some possible usages, is this ok?

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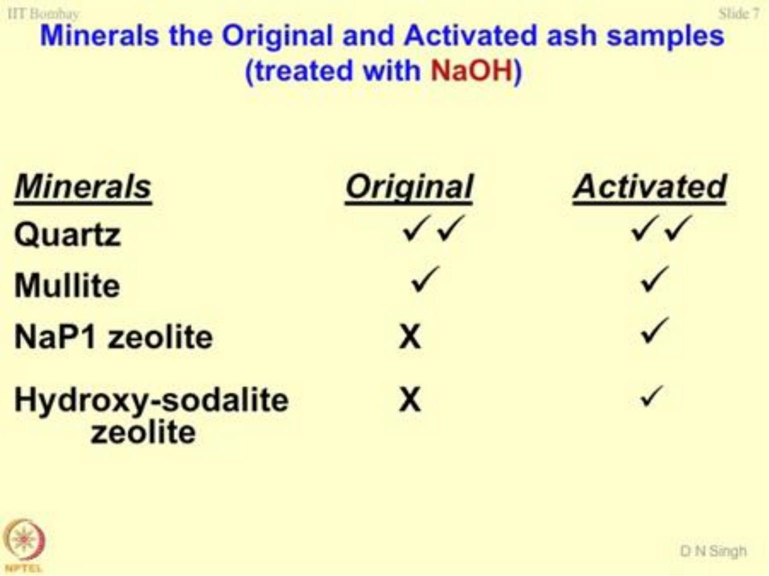

Now, sometimes back I had been discussing about activation of the fly ash samples, if you remember I have shown you some slides where fly ash got converted into a zeolite. So, how would you say you know with lot of in affirmative that something is happened to the material? So, if you compare the XRDs of the original and activated ashes, what you will notice is that quartz is present in both mullite is present in original and activated ash. However, in activated ash there are two more minerals, which are available these are known as nap one zeolite and hydroxy sodalite zeolite. Sometime back I had given you an idea that you can work on SRT. What is SRT?

Student: (Refer Time: 23:38)

Silica retention technology, where you can reduce the silica from the geomaterials and you can increase the components of alumina and iron. Now, any guess how would you do SRT analysis? I am sure you will appreciate this point that if you have different XRD of the samples at different stages of silica reduction, you can make out very easily that whether your process of silica reduction is effective or not that means, the content of quartz and mullite should be decreasing in the activated ash and content of zeolite should be increasing in the activated ash. So, this gives a very broad picture of how material characterization can be done based on XRD analysis. Is this part clear, any questions?

Student: (Refer Time: 24:35)

Oh, ticks mean they are present; cross means not present, absent.

Student: Sir, what's here two ticks meaning?

Oh, two means most prominent. So, two ticks correspond to most prominent phase; one tick corresponds to presence; cross corresponds to absent, and this corresponds to presence only. So, this type of treatment was with the help of sodium hydroxide.

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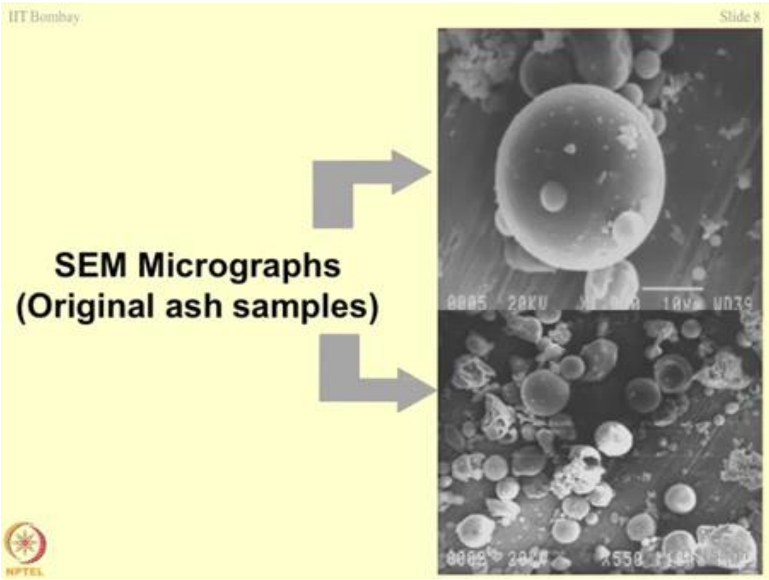

The second characterization scheme which is normally used is scanning electron microscope or microscopy. These are the scanning electron micrographs of original fly ash sample, where you can see perfect balls or spheres which are present. There are some cenospheres, hollow spheres, broken spheres which are present in the ash, is interesting word of fly ash you know. So, the more and more research you do more you get involved with this, difficult to come out of it. At the same time, you may find lot of carbonates also present you see.

So, this type of analysis takes lot of time, you have to sit down patiently, and study the micro graph and see what type of information you can get from this material, so that you can use it accordingly. One more thing you must be noticing there that some numbers are written, here is not like this number. So, this corresponds to 550 times magnification. This is 20 kV, that means, the energy of the light which is being you know thrown on it is 20 kV, and then there will be line. So, this line corresponds to 10µm scale. So, this much information you get. So, if I use this scale, I can find out the size of the particles or the grains which are present in fly ash. Is this ok? It is very scientific. So, you can have more and more information coming out of this, interestingly if you are using an electron probe, you must have used I am sure in geology. Have you used it? So, what happens if I use electron probe along with SEM?

Student: (Refer Time: 27:06)

Exactly, I can find out or I can ascertain what is the elemental composition of each particle just by using some electron probe so.

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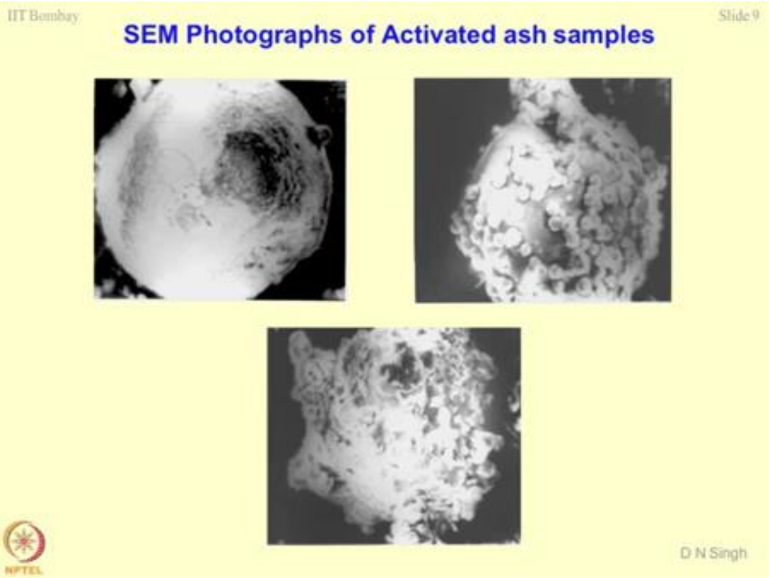

Well, this is the photo photograph which I have already shown you of the activated sample of the ash where some growth is taking place. So, this was also ascertained with the help of scanning electron microscope. We could not do its electron probing because that time it was not available otherwise, we could have estimated the elemental composition of the coating on each particle that could have been a wonderful thing.

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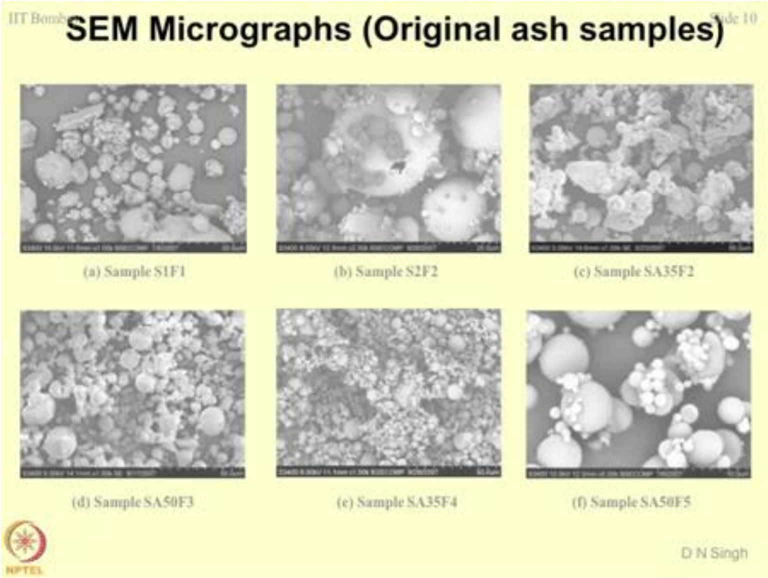

Some of the typical SEM micrographs which I thought I will show you in this lecture of different fly ashes are you do; can you see a typical shape over here. So, sometimes you have either a medullar type of a structure or flaky structures also available in the fly ash particles. This is a very interesting photograph. Can you make out something from here?

Student: (Refer Time: 28:24)

Apart from that? I hope the picture is clear on this screen. Doesn't they look like a nest inside you have eggs being laid by a bird. You can see small, small particles in trapped in this nest. Is it not? So, these are peculiar type of a structure which you find in some of the ashes which are known as claroshperes. So, these are the broken spheres in which there is a particle of ash got in trapped at the time of formation. So, the question is how ashes form is nothing but the at very high temperature the ash particles might have got molten or melt, it trapped lot of particles inside, it has got frozen.

Now, geologist will use this type of slides for finding out the composition of different minerals which are present in the rocks, which is known as spectrographic examination. So, you hope for thin slides and find out the mineralogical composition of the rock mass. You can find different type of structures here, you know some big units' small perfect spherical units, small-small particles of different shapes and so on.

Look at this it is a beautiful assembly of particles is it not. Now, this was artificially created by Dr. Shantha Kumar, the person who finishes Ph.D. recently. I will show you why we were trying to do this type of growth of the particles on the fly ash. I hope you will appreciate this. This is intentional see what we are doing is the arrangements of the finer particles on bigger particles. So, bigger particles are being used as seat. And on this seat, we are growing something ok. Can you use this type of system in geotechnical application?

Student: (Refer Time: 30:42) pores.

Yes, filters, where you can go something in the micro pores. So, what happens? The entire probes get locked, and then you get the best possible durability.

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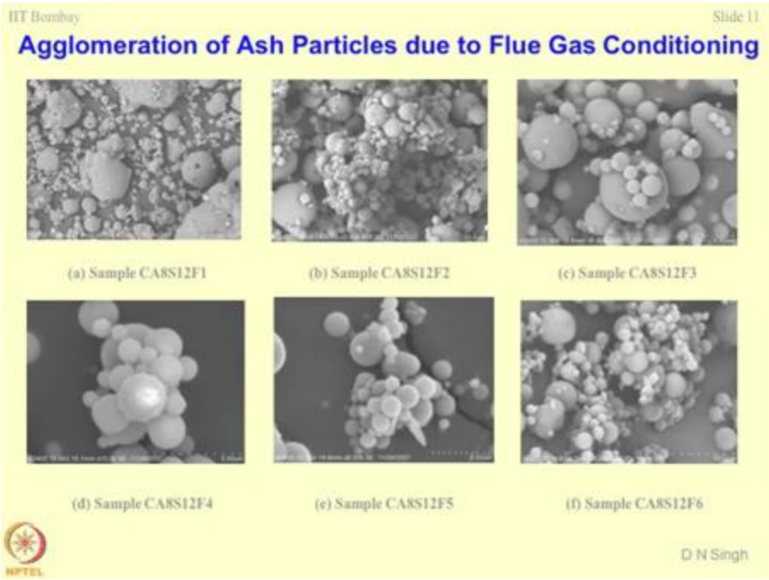

Actually, he was working on agglomeration of ash particles due to flue gas conditioning. It is a new concept in which we worked sometimes back. What we wanted to do is we wanted to create a block of the ash particles for improving the precipitation process in the boiler units, so that no fly ash particles should go into the environment.

Now, this is what is known as SPM reduction, suspended particulate material reduction in the atmosphere. So, if you are going to work in a power plant, nobody is going to allow you to throw the emission or the fly ash into the environment. So, this is where there was a need to develop a technology by which we can capture all the finest possible, clear bone, ash particles and precipitate them. So, this is how the agglomeration was achieved with the help of dosing the gas, the flue gas which comes out of the chimney with the help of ammonia and sulphur trioxide.

So, this is known as ammonia conditioning and sulphur trioxide conditioning. And then you can form different type of structures you know what happens the moment this type of flock is formed, it will precipitate down easily in the boiler units and nothing will go into the environment. I hope you will appreciate that it is a very interesting idea, where you are capturing the finest possible particles making a matrix; it becomes big in size and ultimately settles down in the boiler unit itself or the ash collection unit. Just wanted to show you how agglomeration of the particles can be studied with the help of advanced techniques like scan electron microscopy.

So, you generate a hypothesis, and you can prove your hypothesis by going into the microstructure of the material. Curtesy, advance technology which is present is there. Like 7, 8 years back, it was not really so possible. Here you can see lot of you know ammonium carbonate which is getting formed, and the white colour and the grey colours are of the ash particles. So, again you can get lot of information particularly if you are using the electron probe to determine the composition of each particle, and what type of substances getting formed during the process. Any questions here, any question?

Student: (Refer Time: 33:56)

Sorry, what?

Student: (Refer Time: 33:59)

What extent would be the extent of agglomeration that is what your question is. Well, we could not quantify the agglomeration process. What we have studied is how ultimately the agglomeration is taking place, but yes, your question is very valid. And my guess is that agglomeration phenomena will depend upon lot of thing particularly the surface charge, then the size of the particles, then size the type of the conditioning agent which is being used, the structure of the ash particles and so on. So, if you do a very systematic study, you can define what is the uptake capacity of the ash particles; that means, how many finer particles each ash particle will uptake on itself. So, this requires lot of fundamental study.

Student: (Refer Time: 34:57)

Please be louder.

Student: (Refer Time: 35:00)

Good, this is again a very good question on which we have published two international general papers. I wish you would have asked this question 3 years back. We have shown that there will not be any difference in the ash properties. So, this paper is going to come in the ac general of materials is already in the print, where we have shown very systematically. There was a question asked by the users, they were not collecting ashes from the power plants, because their hunch was you are conditioning them with some chemicals and ultimately the properties may change and these properties may not allow this material to go into the construction materials.

So, we have shown that first of all the dosing which is done is very, very small in terms of its magnitude or amount. Another thing is the whole concept of dosing ashes, you just want this agglomeration to takes place, no mineralogical alteration is going to take place, no chemical composition is going to change, and no other properties are going to change. Interestingly, if you put this material in water again disintegration takes place and all the particles will disintegrate. So, it is a sort of a temporary system by which you are trapping the particles by making a bigger flock.

Because of this reason many power plants are unable to sell their fly ash, because at on one side that they are using flue gas conditioning to trap most of the fly ash which as which is trying to go into the environment, but then on the second side the question was whether this ash should be suitable for construction material or not. So, it is a real environmental engineering problem and soil engineering problem where lot of consequences are seen it is ok. Any other doubt?

Student: (Refer Time: 37:20)

Sorry, please speak loudly.

Student: (Refer Time: 37:25)

Yeah, this is what is known as flue gas conditioning; that means, you are adding either sulphur trioxide or ammonia in the gas fumes or the stack of the chimneys. So, this gas is responsible for creating a sort of a physical alteration, and depending upon the surface area and the surface property of the fly ash particles, this type of process will take place. So, lot of electrostatic concepts involved in this and lot of you know chemical concepts are involved in this. So, truly speaking this have problems are studied by chemical engineers. But because our interest in fly ash and its application in construction material, we entered into this field.

A good study would be you do this type studies using SEM, and then trap the fly ash which is going out of the stacks or the chimneys, and then match the results of the two. So, what we will notice is there will be a drop in the particle sizes which are emitting into the environment because of this process. So, both of them are going to compliment to each other. And this way you can show the efficiency of the traps or the back filters normally which are designed for safe guarding the environment against emission of pollutants in the environment. It is a big technology; right now, only one company in the country has a technology available with them.

So, they design back filters for different emission units for cement plants, fly ash, power thermal power plants and so on. It is a good research idea. And then other question would be how to increase the efficiency of agglomeration further, because there is a limitation to this method, so that is where you have to study the effect of electrical field and chemical field put together on the dynamics of the ash particles in a electrostatic precipitator all right. So, this type of modelling also should be done.

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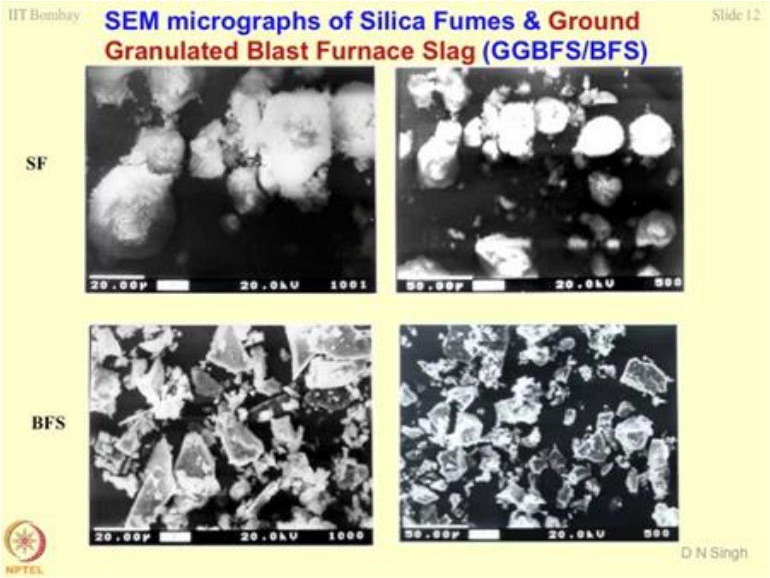

Well, just to give you some feeling of we have been talking about silica fume and GGBFS how do they look like. So, this is a particle of silica fume. What is your observation here, how do they look like? If you remember I had said that silica fume is a highly carcinogenic material, it gets air bone very easily. Why it is so? Look at the shape of the particles.

Student: Its surface area is very high (Refer Time: 40:25)

Its surface area is very high, that is right and you have lot of furze.

Student: (Refer Time: 40:32)

Furze, you know which make it very light, because here interacting in this system is much more, so specific gravity is very very small very less 0.6, 0.7. And because of very high surface area if this thing goes in the lungs, what we will do? It will absorb more and more moisture from the lung and that portion of the body or the lung will become cancerous.

Now, the second group of figures these two are called GGBFS sometimes is also known as BFS that is blast furnace slag. The full form of GGBFS is ground granulated blast furnace slag. Ground means you are basically making it a powder in the granulated form is blast furnace slag. So, this is how this slag looks like. They are all sorts of a regular shapes, but very highly reactive material. Why it should be so reactive?

Student: (Refer Time: 41:41)

Go back to XRD.

Student: Amorphous form.

Amorphous form that is right plus another thing when we talk about chemical composition then I will show you that why they are so reactive. Is it ok?