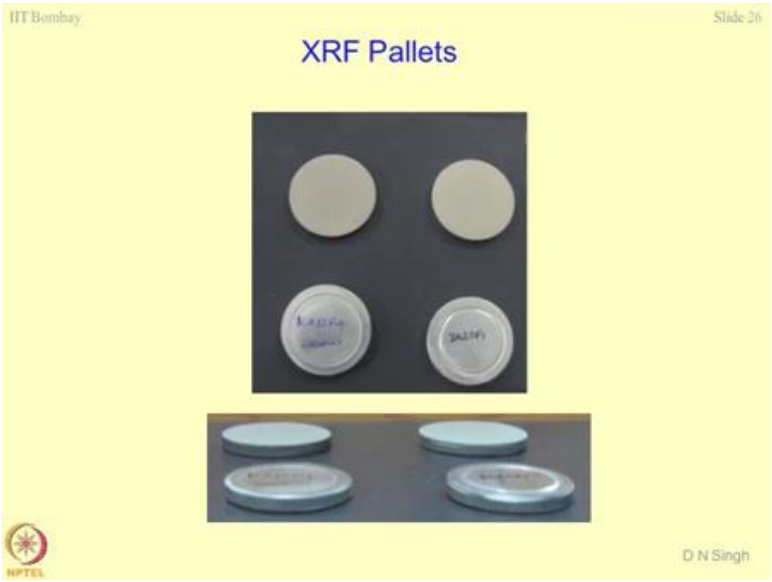
Environmental Geotechnics
Prof. D. N. Singh
Department of Civil Engineering
India Institute of Technology, Bombay

Lecture - 23
Geomaterial characterization -3 (Chemical Characterization)

(Refer Slide Time: 00:15)

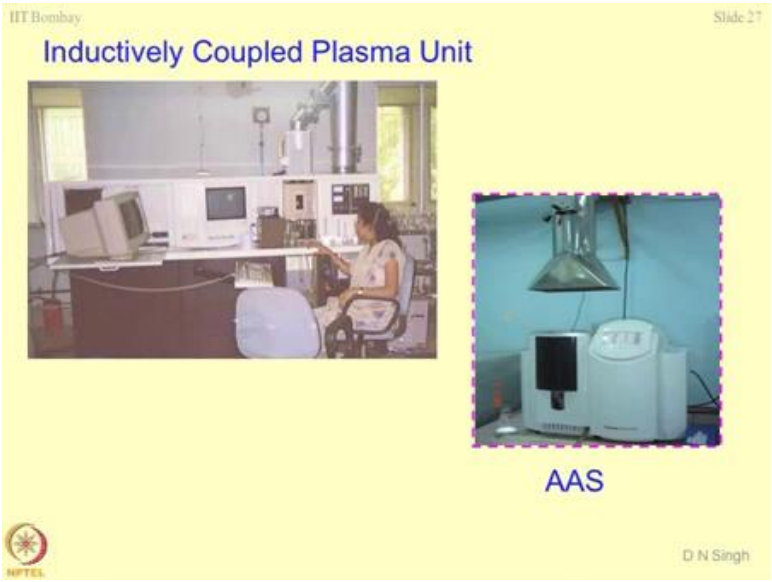
Let us talk about a bit of chemical characterization. So, most of the time chemical characterization is done to understand the chemical properties of the material, where XRF is done X-ray fluorescence technique, this is a setup which is XRF, do not think that is washing machine. It is a XRF setup from our IIT Bombay at SAIF, very expensive equipment cost than more than I think 2-2.5 crores. Then second unit is ICP - inductively coupled plasma unit, which gives you the concentration of trace elements. Then pH value we study, cation exchange capacity and then pore solution analysis.

(Refer Slide Time: 01:09)



So, these are the pallets of X-ray. You can imagine a 0.5cm thick aluminum disk which is normally used. And half of this disk is filled up with cellulose state and the material for which chemical composition is to be obtained. And this makes a bond, you cover it apply some load, so that becomes a pallet and this pallet in this form goes in to the X-ray machine to get the fractions of oxides which are present in it.

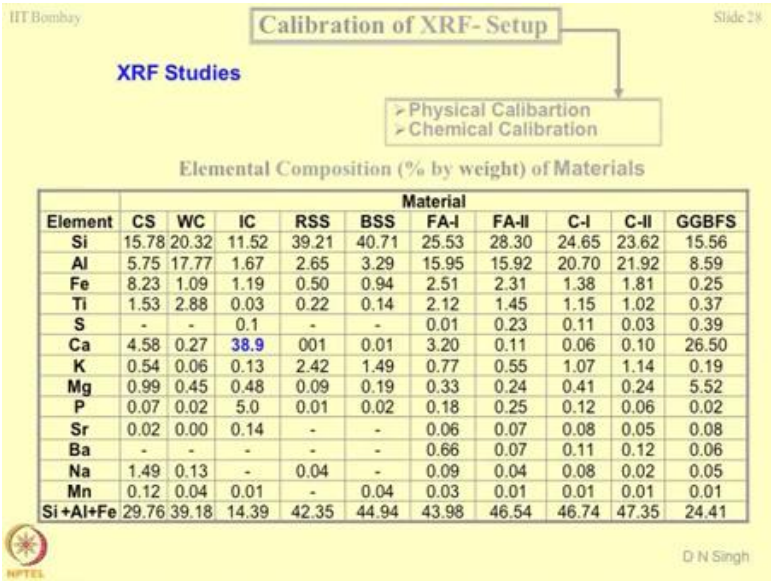
(Refer Slide Time: 01:45)



Now, this is the inductively coupled plasma unit, where you can find out the trace elements which are present in the soil mass up to the ppt level - parts for trillion (i.e., 1 in

10^{12}). So, this instrument which is on our lab can be used up to ppb (i.e., 1 in 10^9) or ppm (i.e., 1 in 10^6). But then this equipment can be used for finding out the concentration up to ppt level - parts for trillion.

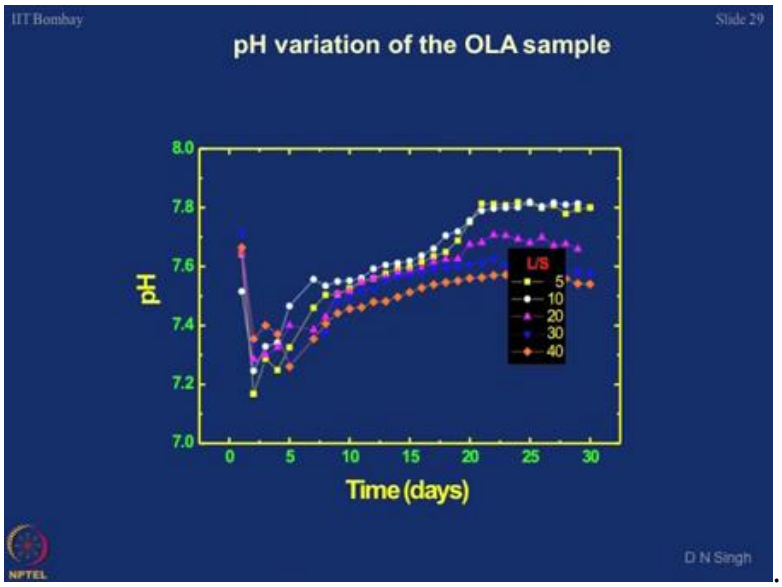
(Refer Slide Time: 02:13)



	Material									
Element	CS	WC	IC	RSS	BSS	FA-I	FA-II	C-I	C-II	GGBFS
Si	15.78	20.32	11.52	39.21	40.71	25.53	28.30	24.65	23.62	15.56
Al	5.75	17.77	1.67	2.65	3.29	15.95	15.92	20.70	21.92	8.59
Fe	8.23	1.09	1.19	0.50	0.94	2.51	2.31	1.38	1.81	0.25
Ti	1.53	2.88	0.03	0.22	0.14	2.12	1.45	1.15	1.02	0.37
S	-	-	0.1	-	-	0.01	0.23	0.11	0.03	0.39
Ca	4.58	0.27	38.9	001	0.01	3.20	0.11	0.06	0.10	26.50
K	0.54	0.06	0.13	2.42	1.49	0.77	0.55	1.07	1.14	0.19
Mg	0.99	0.45	0.48	0.09	0.19	0.33	0.24	0.41	0.24	5.52
P	0.07	0.02	5.0	0.01	0.02	0.18	0.25	0.12	0.06	0.02
Sr	0.02	0.00	0.14	-	-	0.06	0.07	0.08	0.05	0.08
Ba	-	-	-	-	-	0.66	0.07	0.11	0.12	0.06
Na	1.49	0.13	-	0.04	-	0.09	0.04	0.08	0.02	0.05
Mn	0.12	0.04	0.01	-	0.04	0.03	0.01	0.01	0.01	0.01
Si+Al+Fe	29.76	39.18	14.39	42.35	44.94	43.98	46.54	46.74	47.35	24.41

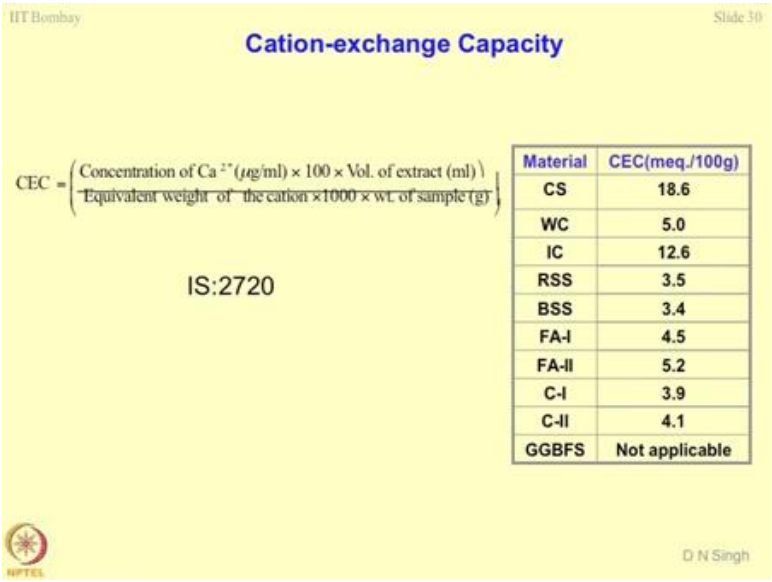
Now, these of the results of different materials which have been analyzed. You will notice that the calcium is present in the choke sample and 26.5% is present in GGBFS and in your normal white clay the calcium is only insignificant 0.27. The fly ashes are also very poor 0.11 except for one type of fly ash which might be a different type of source of whole. So, this is the reason why your GGBFS is very active. It has more calcium. This is what is known as elemental analysis of different oxides.

(Refer Slide Time: 02:57)



Well, this type of study becomes very important to see how the pH of the material changes over a period of time. So, you have to establish past whether the soil or the geomaterial is acidic or basic and how these acidity or basicity changes over a period of time. So, this type of a study is a normally done particularly by people who are in associated with concrete technology, that how easily the system becomes acidic or basic. The common sense says that you cannot use the material which is acidic in making concrete. So, you may see the alteration in the response of the material, it may dip then again pick up in terms of a pH. So, these types of studies are done to study the response of the material over a pro long duration.

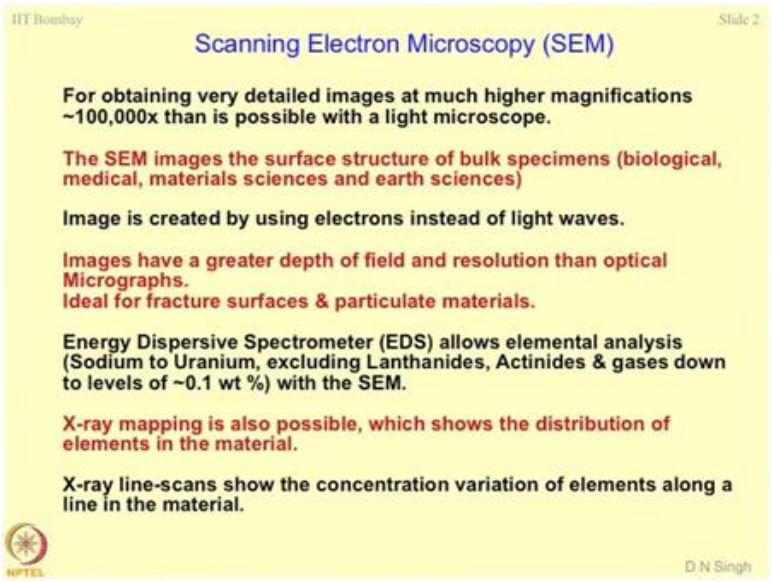
(Refer Slide Time: 03:47)



And the last is scheme of characterization of the materials for their chemical property this is known as cation exchange capacity. So, by definition cation exchange capacity is the concentration of calcium ions which is present in certain volume of the extract divided by the equivalent weight of the cations. Now, these cations are nothing but the cations which are replaced by calcium ions. So, that is a reason why we call it as a cation exchange capacity, how easily the cations can be exchange from the soil mass by the contaminant. So, here you have to treat the soil sample repeatedly by sodium hydroxide (NaOH) and calcium hydroxide ($Ca(OH)_2$). And then see by every trail how many calcium ions are replacing sodium ions.

So, this is a sort of a reactivity. So, for different materials we will notice that the cations exchange capacity its units have m.eq. for 100 grams. This varies and you will find that for different type of soils, the values are quite less. However, for blast furnace slag you cannot do this as analysis because the moment you add water it forms the thick gel it sets. So, this type of test cannot be done.

(Refer Slide Time: 04:59)



The basic idea of doing SEM is to get a very detailed idea about the material. Particularly various stops by the simple optical imaging this is where SEM begins or this is the domain of SEM begins. So, for obtaining very detailed images at much higher magnifications and resolutions approximately 10^5 times if you want to study any sample or a specimen then you can adopt for a SEM and this is an improvement over simple like microscopy.

The beauty is that SEM images the surface the structures of bulk specimens. And nowadays, SEM analysis becoming very common and different types of studies related to biological sciences, particularly tissue cultured if you are interested in study in medical sciences, material sciences, earth sciences and in civil engineering also we are using it, because we treat geomaterial as a natural material or manmade material.

So, what is essentially done here is that you create a image using electrons instead of the light waves. So, in the simple photography you use light waves for capturing a photograph; however, in SEM we use electrons of certain energy. So, basically the entire SEM a spectroscopy or microscopy is based on the fact that you use electrons of different energy and their scattering. The beauty of this technique is that images have a greater depth of field and resolution then optical micrographs. And that is why it becomes an ideal technique for studying the fractures surfaces or the particulate material

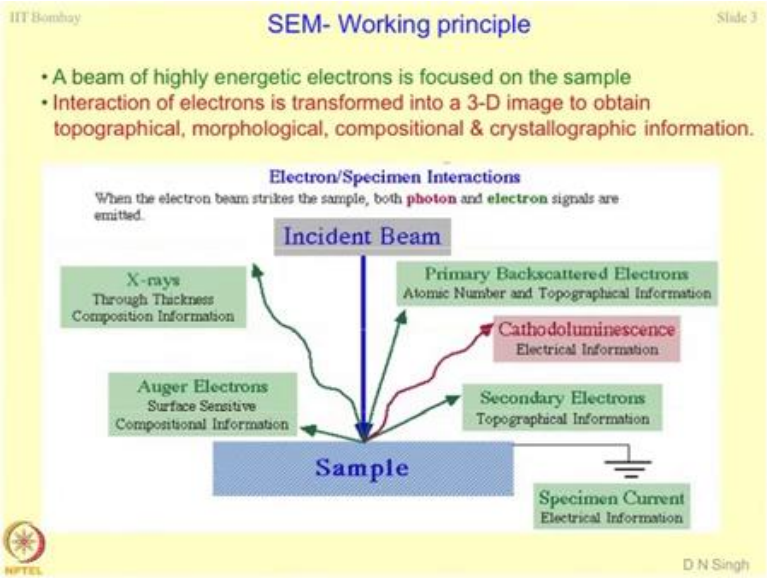
is it not, where you have more of particles arrangement which you want to study and the fracture surfaces are irregular, not very shining, broken and so on.

Now, this is the recent development where EDS is normally used this is known as Energy Dispersive Spectrometer. And when you add this along with SEM so this becomes EDS-SEM (EDAX), so this allows elemental analysis also and that means, you can do the analysis of sodium to uranium, how were you cannot study lanthanides, actinides and the gases if there happening to be less than 0.1 percentage of the weight. So, this is the beauty of the technique another good technique another good implementation of this technique is the X-ray mapping is also possible in SEM.

And which show the distribution of elements in a material if you want to do the elemental analysis SEM becomes a very good tool. Remember in X-ray also you have done elemental analysis X-ray florescence technique, where you can get the composition of oxides by weight this is a chemical characterization of the material. Another, interesting point is that it can do line scanning, so X-ray line scans we will show you the concentration of the elements along a line of the in the material.

So, how the elemental composition is varying from one point to another point can also be studied. I would not go much in details of these topics because then it becomes a you know subject itself. But I am just giving you again some idea about what SEM is what are the beauties of this technique and what are the limitations.

(Refer Slide Time: 08:45)

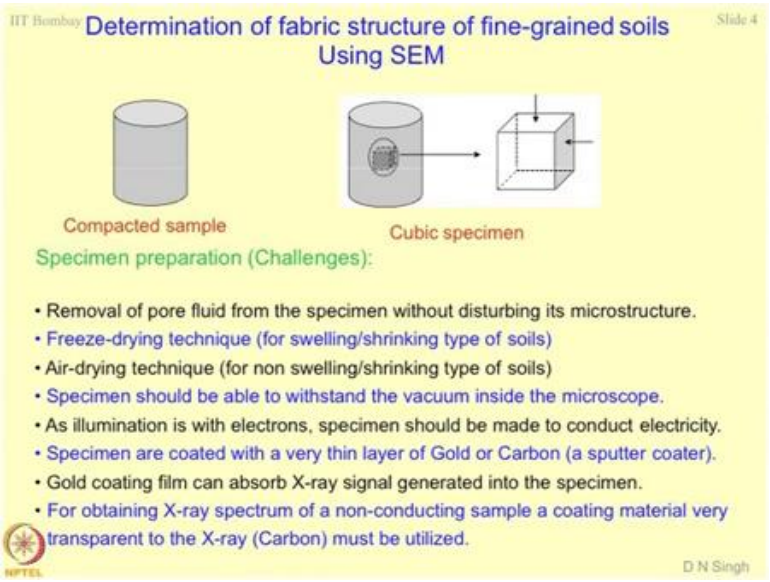


What is the working principle? The working principle is you have a beam of energetic electrons, which is falling on the sample sometimes you call it has a specimen also. And then interpretation is done by using again collection of X-ray beams or the electrons, which are getting generated in the system. And what we have study essentially is the interaction of the electrons and this transformation is done on a 3D image to obtain a topographical, morphological, compositional and crystallographic information.

So, this is the beauty of this technique. This figure shows you how sample is bombarded with an incident beam of electron, so basically when the electron beam is strikes the sample both photon and electron signals are emitted. Now, what is that you want to collect and what is that you want to analyze is an art, so if you consider this as a sample on which there is a beam of electrons falling and then there is a scattering, so you can have a X-rays, you can have other electrons, you can have primary back scattered electrons, which you want to collect for our analysis and this will depend upon the atomic number and topographical information.

Then we can have cathodoluminescence, that means how electrical information can be obtain from this type of a finger print of the material. And then if you can collect secondary electrons, you can get the topographic information. So, this is how you can do 3D topography of the system by using a scanning electron microscopy. This is a general outline of how SEM works by takes times to analyze the samples and sample preparation, so this I will be discussing in the subsequent slides.

(Refer Slide Time: 10:41)



Now, this is a topic on which Suchith is working. Determination of fabric structure of fine-grain soils using SEM, so this is one of the applications of the scanning electron microscopy. And he has gone one step further where he is amalgamating SEM with MIP that is Mercury Intrusion Porosimetry, so most of the slides I have taken from his presentations from during the different stages.

Now, first thing is you create a sample of compacted soil, so this is cylindrical tri axial sample and I want to let us understand what is the fabric structure. When we talk about fabric structure basically, this is a grain structure and their arrangement which we are interested in finding out. I hope that you will appreciate that this type of studies cannot be done here by simple optical photography.

In previous lecture, I have talked about 2D con-focal microscopy and 3D photography, is it not surface photography. So, those techniques cannot be used here those techniques are used for morphological examination, so this is the model which has been developed by Suchith. He extrudes the sample, which is known as the cubic specimen from the sample and then his studies the orientation of the grains or the fabric structure in the two perpendicular planes. Now, what are the challenges which are associated with the specimen preparation? We call this as specimen small content and this is a sample of the soil and it is understood that this specimen happens to be representative of the entire sample.

So, one of the topics on which we are working right now is we are trying to simulate nature how sedimentation takes place in oceans. So, you can imagine if you have a sample of 6 centimeter length based on the sedimentation process, how grain structure is getting formed can be studied by taking out sample under in situ conditions as a form of a UDS (i.e., undisturbed sample) and then taking out this a specimen and analyzing it for SEM. So, once you have taken out this cubic specimen, you have to answer few challenges or you have to gear-up for handling these challenges. The first challenge is this sample is going to have lot of pore fluids is it not.

So, how to get rid of this pore fluid? So, the first challenge is removal of the pore fluid from the specimen without disturbing its microstructure. Do you think that there is any technique by which you can remove the pore structure from the soil mass? Apart from the techniques, which should be adopted for this type of a work. The most ideal situation would be if I can squeeze the sample just by applying some pressure, which is what is done in a pressure membrane extractor. If you remember the other day when you came to the lab had shown you a device by which you can extract pore solution, but then it will be very difficult to maintain the microstructure if you are applying external pressure.

So, this is where we go for freeze drying technique, where shown you liquid nitrogen gas, so if you dip the sample in this liquid nitrogen you can get it of the liquid phase and the entire structure gets frozen. Now, unfortunately this technique is not suitable for all type of soils and this is very much suitable for the soils, which are swelling and shrinking type of the soils. But suppose if you are dealing with the soils which are ordinary soils, which are very passive materials, so this is where you can use air drying technique you can dry the sample in the air. And you can remove the pore solution, which is present in the soil mass.

Another challenge is that a specimen should be able to with stand the vacuum, which is present in the microscope that means it should be stiff enough and should not get broken. Now, each one deals with development of a technique, so that is why I said that SEM is also a very intricate method of doing the analysis. Another challenge is that as illumination is with electrons, specimen should be made to conduct electricity, but you will agree with the fact that most of the materials in are in the powder form and their rock soils including soil, which are not good conductor of electricity, so that means you have to give some treatment to the a specimen therefore, you can do SEM analysis.

So, this is where some coating is to be done. And this coating is either of gold or of carbon depending upon the requirements, so these specimens are coated with a thin layer of gold or carbon, which is also known as the sputter coating or a coater is name of the machine. When should you use a gold thin film or when should you use a carbon thin film it all depends upon what is that you want to do. So, gold coating film will absorb X-ray signals generated into the specimen. And for obtaining access spectrum of non conducting sample coating material very transparent to the X-ray like carbon should be used.

So, there could be a situation when you are taking SEM photograph and the sample, may not show any response. So, this is your you have to switch over from gold to normally carbon coating, so again this comes from your experience and the type of material which you are handling. This is okay? You wanted these details.

(Refer Time: 16:45).

Geotechnical engineering is an art. You know most of the experiments which geotechnical engineers do they are done as an artist, what this indicates is making a sample of 6cm length may take you 2-3 days, taking a specimen out of the sample may take you 1 week. You know it is a very slow process and very have to very patient that is only answer is this correct Suchith. You would like to add something.

(Refer Time: 17:20)

That is what I say it is not a challenge, it is a temperament. Somebody may do this test. Somebody may not do this test. You may not be able to take out the sample out of a specimen out of a sample, so it is basically a temperament nothing else. So, when you do direct shear test, the tendency is you pour the sand and just fill it and in 2min your sample is ready. Truly, speaking and we work on our direct shear test and the samples I mean take at least 18-20 hours to make a direct shear sample. We just go by grain by grain and by grain and grain and grain and so on. So, we arrange the sample based on the granular unit. You just do not pore the sand and do the test like this, so that is what I said that our subject is more of an art.

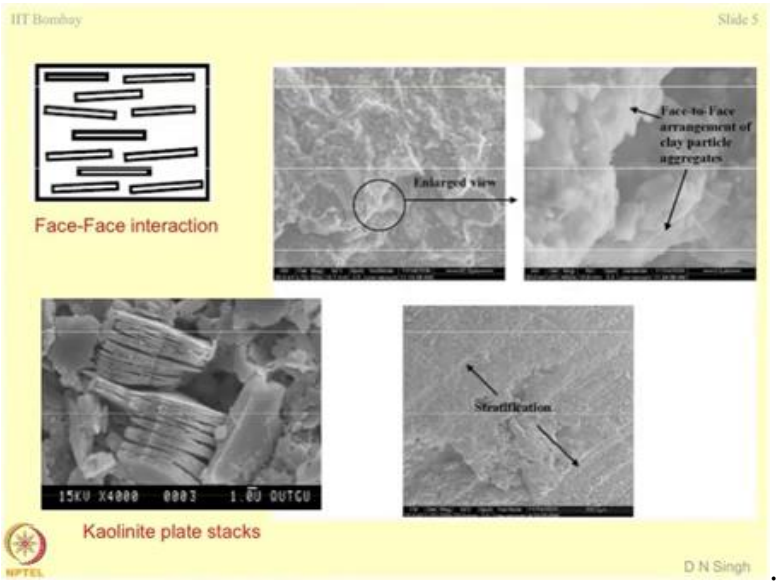
And most of the philosophies are you know included in the subject; it is not mathematics where you put a + b = c. It is a science as an art, but yes, your question is very correct.

Challenge is there, but then I have not included in this list, but to make you happy we can say yes, the first point should be how a specimen should be retrieved out of the soil sample. But that is not a very big deal I suppose or it is so you can answer his point. So that is why your technical engineering is you know too much research oriented and one should have a temperament to do research; research cannot be forced.

Student: Another answer to your question will be why means these challenges not included in this? So, the thing is basically in this study. We wanted to know the structure of the you know particles or the arrangement of the particles in two different directions ok. So that is why this is the; this is the need for this particular study. So, generally whenever you do the SEM is you need not be very you know about the taking out the samples means just you are interested in the you know top most surface, but here I am I was interested in not only the top most surface or the horizontal surface, I was interested in the perpendicular surface also, so that is why I took lot of time to you know cut the sample with the help of a knife properly.

And then keep it for the drying purpose. So, that is why this challenge is I would say this is for this particular study, but if you want to know general about the specimen. Just we can take out the sample and use just cut some part of it and keep it under microscope. So, like in case of let us say polymers are concrete, but even concrete samples are also very tough to you know extrude from a sample, but then you have to device away. You cannot say that this cannot be done ok. Are you satisfied or no? say that this cannot be done all right. So, these are the challenges and of course, what you say Jan that can also be included in the list?

(Refer Slide Time: 20:41)



Well, this is the typical face to face interaction which normally you know is depicted in the text books and you have been studying in undergraduate, but did you other question to see this laterally, where whether this structure really exist or not. So, I will try to show what Suchith is obtained in his studies.

Now, this is what actually we got by doing a SEM analysis of the sample here you can see this platelet. And this is just sitting above another plate can we appreciate this is clear or no? Now, if you in last this view this is what you get, so this is one platelet and there are so many other plates you know, which are underlined these are sheets. So, this is how you say face to face contact I will show another photography, where it becomes much easier to visualize is the figure clear or no? Little bit hazy contrast is less ok.

So, this also a problem with the researcher they get too much by their own findings. So, we start seeing everything in our own doings you know others may not observe, but you can see this stratification over here. So, these are the stratification you know stress formation a layer wise is this clear Suchith or no? You can add something if you want.

Student: basically, the first two figures. Now, these photographs are taken from the top and the third figure is taken from the sides.

So, if you understand now from the this left figure face to face interaction that if I take it from the top, I can get you know the layers ok. And if I see from the sides, I can get those stratifications.

This one is clear; you know you can see.

Yes.

This.

Yeah.

Sheet lying over the other sheets.

Student: So basically, in SEM what I have to do first of all I have to keep the sample in one direction and to take the photographs and then just change its face.

Yeah.

And again, I will take the photo.

Yes

This is a much better picture of kaolin. So here you can see the facts of the sheets of kaolin. You know so this is exactly face to face arrangement of the grains of kaolin. Of course, this is at 4000 magnification. So, this proves the granular structure or the particulate structure of the soil mass. Another, reason could be that you are working on marine clays I suppose.

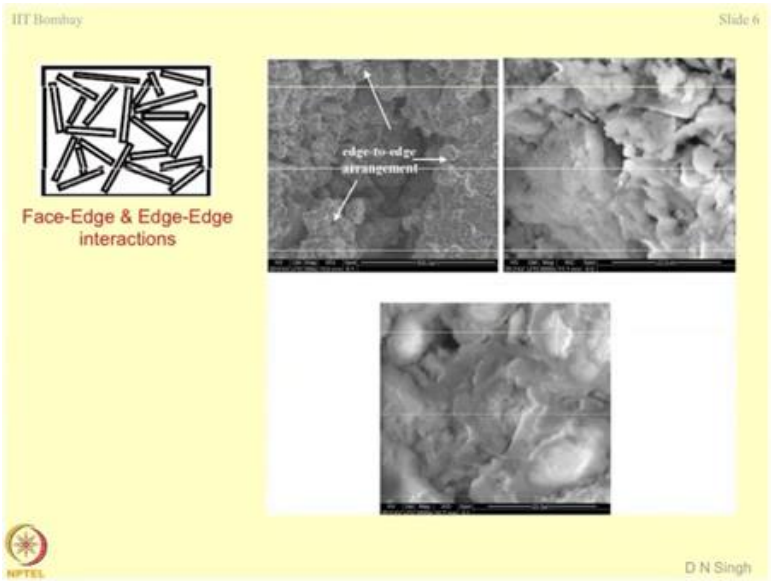
So, marine clays will show you lot of you know impurities and because of that there may be some haze however this was a pure mineral on which you are perform the tests. So, these are the sheeting's or the kaolin which are very clear. And you know now it gives you a feel of how face to face interaction can be really defined and what causes this type of interaction ultimately how material properties are going to get altered. Now, my question to you is why these studies are required? Till now we have been doing only compaction curve is it not without bothering about what really is happening to the soil mass, when you move on from dry of optimum to the wet of optimum crossing over the OMC.

So, for that you have to wait for some time and when he present his results in his annual progress seminar and then you should come and see that there is a transition of the orientation of the grains from dry to the wet of optimum and here literally captured all this things beautifully. And then he is trying to give assign a electrical number associated to the arrangement of the grains and that number tells you what type of fabric structure is present in the soil mass. So, this is this Ph.D. topic, how much time normally you take to complete one sample analysis.

Student: Sir, preparation of sample take at least you know 1 day or half day means for each specimen. I would say and then taking the photographs and analyzing it. So, at least it takes a week for 4 to 5 samples.

As gives you fair idea about the grain structure.

(Refer Slide Time: 25:21)



Now, look at the second type of structure which we have been studying in under graduate. Face to edge and edge to edge interactions is it not. This is what how do you call this as the structure, flocculated structure. Truly, speaking this is really is, this a flocculated structure or not. How would you define this structure, this is a dispersed structure. Truly, speak is not a real flock it is a cardoon structure. It is a pack of cards. Now, if you look at the micrographs, you have a grain here and you have a grain here. So, this is you know edge to edges interact arrangement, is this clear? Suchith is this clear on the photographs?

Student: And again, now if you see these three photographs 1, 2, 3, first one it is taken on the dry of optimum on the compaction curve ok. The second one is you know near the optimum water contain and the next one is weight of optimum. So, you can see the grain structure or the fabric structure changes as we move on the compaction curve and the basically count why the particles are getting compacted or why we are getting dry density more as we go for higher water content and up to certain water content. So, it is because the particle packing or the density of the particles is increasing, so that is a reason.

How much time it takes to analyze this photograph? Suppose if this photograph is given to you how much time normally takes to analyze the photograph to make out something out of it?

Student: Sir, basically it depends on the judgment basically and intuition.

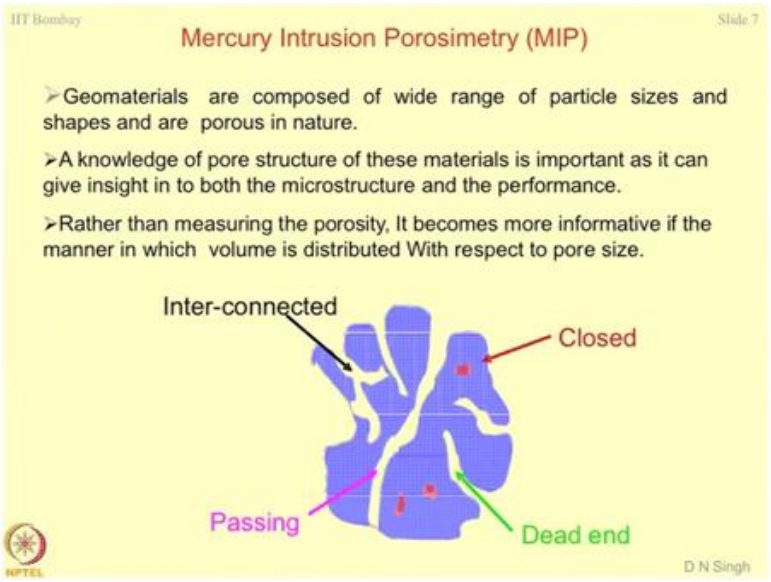
So, given a chance you may not find any interesting mechanism happening here.

Student: I am not able to interpret what is happening now.

So, that is what actually I am telling you that it requires a third eye to observe what is happening here. Look at these two grains you know they are just sitting over the edges is not the flux as compared to the previous photograph where the flux was there. So, if you compare this, this is only edge and edge of the particles magnified to almost 4000 to 5000 magnification. Now, by the time you come over here it has become more amalgamated.

So, this type of studies can be done with the help SEM, so that is beauty of finding of these structures and you know why do we need to study the grain structure, so that we can correlated to the hydraulic conductivity. So, the best way to make models for hydraulic conductivity would be the models which are based on a scanning electron microscopy results and the orientation of the grains, so if you can quantify them, anyway this is of more of research importance. Anything else which you want to add on? SEM, That’s good enough?

(Refer Slide Time: 28:45)



Student: Can I talk about something about limitations of SEM?

Why not.

Student: So, as you said this interpretation of the results is again, I can say it is a challenge. So, how do you observe means how many micrographs till now you have gone through. So, from that you get an idea that how researchers you know the study the SEM micrograph from that you can get that I should go in this direction, I should study each and every particle and its arrangements, so that gives you more feeling.

So, again that interpretation at what magnification you are taking that is that again some matter matters a lot. Because if you are taking photographs at say 500 magnification and 4000 magnification, so same photograph, but at different magnifications they will give you something different information, so that also matters a lot. So, many things are there you know to for interpretation for proper interpretation of the photograph.

It is just like zooming in and zooming out of a picture to get the information of your requirement.