Environmental Geomechanics
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Lecture No. 30
Geomaterial characterization-VI
(Chemical characterization)

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XRF Pallets

So, just to give you an idea about these are the XRF pallets if you get a chance go to the SAIF and you can see how these pallets are done. These are small aluminium pallets, half of them is filled with the adhesive these all standard methods nowadays they will help you. So, half of this is filled up in cellulose, and the rest of the half is filled up with the material for which you are trying to find out the chemical composition.

So after this thing is done, you press the upper portion of the pallet, it becomes a concealed pallet. The whole thing is kept under a UTM of about 5 to 10 tons so that it becomes a homogeneous system between the material which you want to analyze and the cellulose and cellulose dries up later on. So, this palette is inserted into the XRS machine, which I showed you, and then you can do the analysis.

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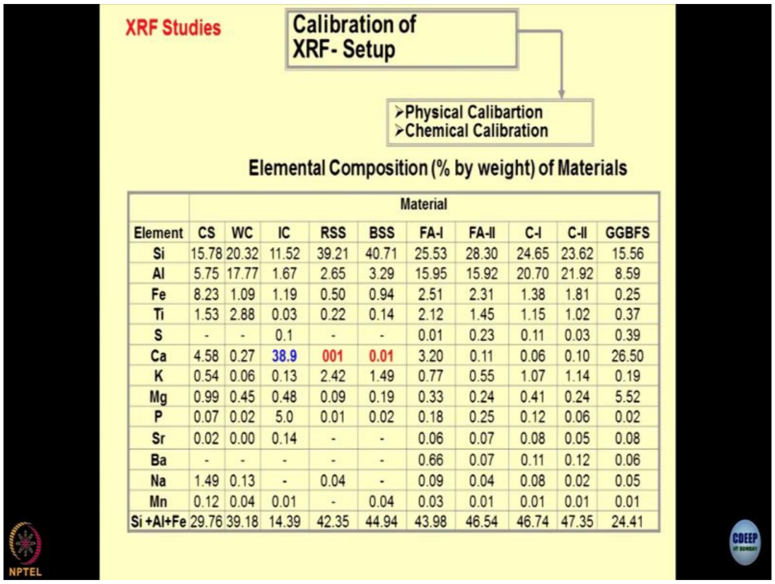

XRF Studies

Calibration of XRF- Setup

- Physical Calibartion
- Chemical Calibration

Elemental Composition (% by weight) of Materials

	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

Just to give you an idea about what type of calibrations are done before you can use the results. There are two types of calibration certain physical and chemical calculus of the machine. Fortunately, chemical calibration is standards. So, these standards you have to buy from the market and you have to take along with you for doing the analysis. As I said, SRF analysis gives you elemental composition. This is the first time we are talking about the elemental composition. This elemental composition could be of soils; this could be of admixture; this could be of any geomaterial.

So, if you look at this, the way we read the value is the percentage by weight of the material. So different type of geomaterials when they were analyzed, these are the results. Any lab will give you these results; the problem starts once the results are in your hands and are able to use that. That is where that entire catch is. And unfortunately, there is no guideline for that because I am sure that even if I write a book on how to read cardiograms.

I am not going to become a cardiologist; you agree a lot of information judgment is required, and mental thinking is required to eliminate the doubts and come to the right point. But some quick analysis, which I wanted to show you, based on the XRF analysis would be calcium is the most important thing, and most of the materials are light. So, if you take Israeli chalk, which I was talking about earlier.

They started the calcium presence is extremely high as compared to any other material. In this series, this is a naturally occurring source of calcium, or if you look at GGBFS, you will have a very high value of calcium at 46.5, which is manmade. So, now, once you have understood the strength of the material, you can utilize it. So, if I require this situation if I want a material for a situation where calcium is required, what I will be doing, I will be adding GGBFS to this.

This is one of the strategies I will not be using this material as a fill material, where more silica is required. So, if you look at this material, sandstones you have a very high amount of silica-alumina, iron and so on. So, depending on which industry you are running, and what is your professional activity, what are your interests? This information can be used some question you are asking a few minutes back, and I said hold on.

Why because I wanted to show you this. So if you are looking for a material which is inert, crystalline, if good fill material for the foundation is should be selecting something which is going to be having a lot of silica into it. This is a poor choice because here silica is not much; this is an aquifer. So if I am in let us industry where I am trying to develop zeolites, yesterday, we were talking about zeolites conversion of silica into zeolitic material.

Normally, we talk about alumina silica ratio. So, if the alumina-silica ratio is of a certain value, then only the material can be zeolitized. So, I have created another application of the material. Are you realizing? So, this is how the strategies have to be created. So coming back to the simple things, issue and if you had alumina-silica and soon, and if this happens to be more than 70% there are some classifications of the fly ashes where these fly ashes are supposed to be inert systems, good film material.

However, it fly ashes do not have alumina, silica iron much around 20 30% but calcium is very high, it becomes a cementitious could be in. So, an industrial waste the moment you do XRF analysis, chances are that about 80 to 90% of the strategy of utilizing it can be created immediately and that is the crux of the discussion. So, just by doing the simple analysis, you can realize what I should be doing. Another question could be if you cannot do something with the material, how to do it, how to augment the properties.

So, all these concepts which are loud are coming, in medical sciences, particularly silica implantation of different types of artificial bones creation. So, from when you bring calcium, there must be a process where I can do segregation of silica from the inner systems. I can precipitate it in a solution; I can make it hyperactive is on. These are different industrial processes.

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Inductively Coupled Plasma (ICP)

Historic

Contemporary

Atomic Absorption Spectrophotometer (AAS)

Gas Chromatograph with high resolution Mass Spectrometer (GC-HRMS)

Just to show you quickly how this ICP unit looks like earlier days, this used to be the situation historic and nowadays everything is very compact. I will show you this is all the contemporary ICP looks like and very compact systems, you can install them in the laboratory and a lot of literature available on ICP and ICPMS.

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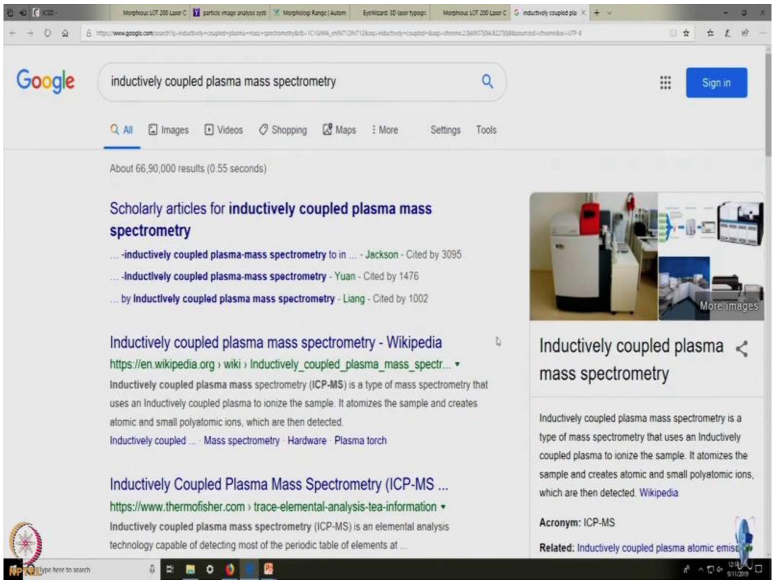

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Inductively coupled plasma mass spectrometry is a type of mass spectrometry that uses an Inductively coupled plasma to ionize the sample. It atomizes the sample and creates atomic and small polyatomic ions, which are then detected. Wikipedia

Acronym: ICP-MS

Related: Inductively coupled plasma atomic emiss

So, when I was dealing with the projects from the atomic industry, and particularly from BARC and atomic Energy Regulatory Board of India, then I used to use ICP and ICPMS quite a lot to detect how many portions of the radioactivity has been solved by the science and how much is released. And so on, these are very interesting gadgets. Atomic absorption spectrophotometer you might have Using your environmental engineering lab to find out the concentration of contaminants up to PPM levels part per million, but when you are looking for parts per billion parts per trillion, then you have to do ICP analysis.

This is all the gas promotor graph looks like. High-resolution mass spectrophotometer. As I said, environmental geomechanics relies heavily on in fact, the practice of environmental geomechanics relies heavily on these gadgets. These are tools which will help you in identifying the problem.

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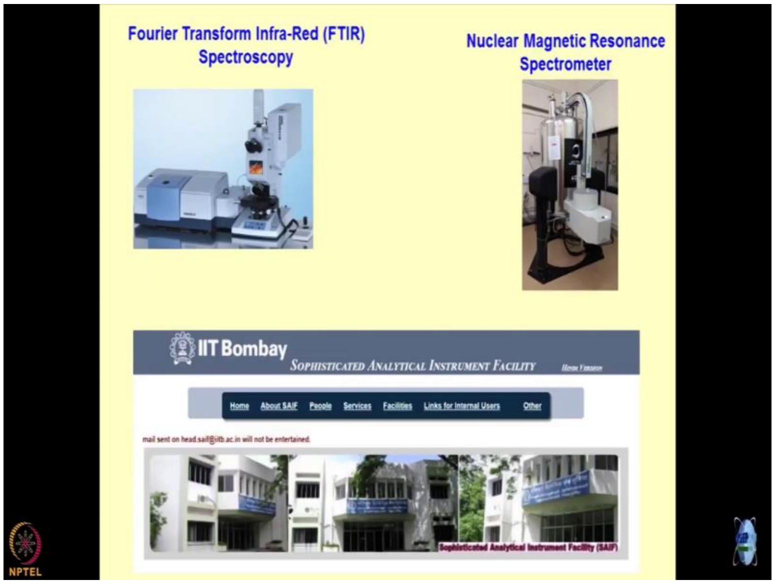

Fourier Transform Infra-Red (FTIR) Spectroscopy

Nuclear Magnetic Resonance Spectrometer

This is how the FTIR looks like this is a Fourier transform infrared spectra for a meter is spectroscopy. And then, NMR. He wanted to do some experiments by using NMR at in one of the universities in France; this is a place where most of these facilities are housed in IIT Bombay. It is known as SAIF, sophisticated analytical instrument facility. And this is worth visiting. But these are the latest development since last 5, 6, 10 years what is going on.

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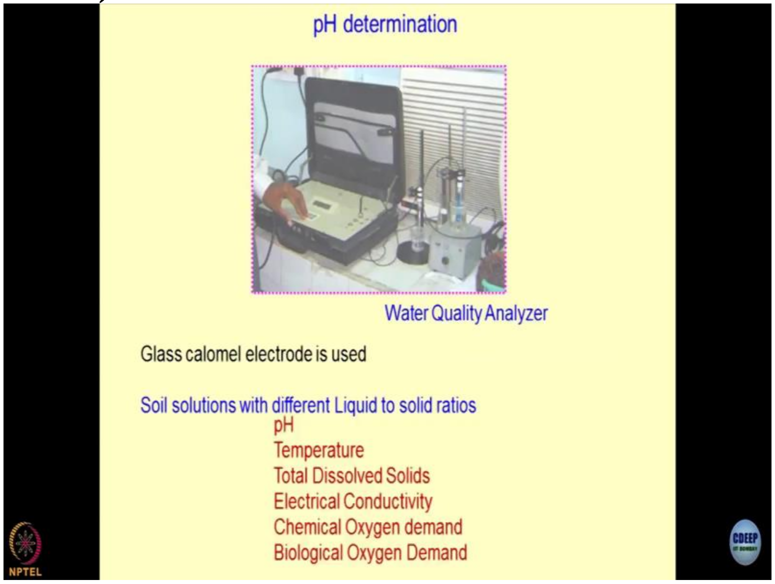

pH determination

Water Quality Analyzer

Glass calomel electrode is used

Soil solutions with different Liquid to solid ratios
- pH
- Temperature
- Total Dissolved Solids
- Electrical Conductivity
- Chemical Oxygen demand
- Biological Oxygen Demand

pH determination emission simple thing, I am sure you must have used it in an environmental engineering lab. We have a water quality analyzer different type of electrodes which can be utilized as glass calomel electrodes you must have used for finding out the pH because then the reaction takes place between the material and the environment, water or any contaminant, we would like to see how pH alters the way the pH of the body changes, similarly, the pH of the system changes soil contaminants system and that becomes very problematic many times.

So most of the industrial byproducts are at different pH values. Either they are highly acidic, or they are highly basic. So the question is, how would you neutralize them how would you create a synergy between the soil and something which is quite aggressive and pH value. So, these type of thoughts people are having, and a lot of research is being done Jasmine is working on a project we are trying to utilize highly basic industrial byproducts the pH is 12 13 by injecting gases into it and this trial we are doing the laboratory so that the whole thing can be injected at one of the heaps in real life.

So, we will have to design a complete piping system in a system you have to force gases to go inside and complete mathematical modelling. So, you must be aware that for determining pH, we dissolve the soils in the water at different L/S ratios, liquid to solid ratio and then the mixture is it and these are the parameters that you can obtain. You can obtain the pH, the temperature, the TDS, total dissolved solids, electrical conductivity and nowadays in our profession. This oxygen demands BOD and CODs are becoming very very important.

You see when I was a student, I never thought that these things are going to be a part of geotechnical engineering. But sometime back I give you an example of my student's thesis Dr Sushmitha Sharma over down the sediments from the different water bodies and sedimentation tanks, which had apart from chemical activity, the pathogenic activity also. So there we realized that COD, BOD is going to be very important for the sediments also.

Because they add up to the kinetics of the system. Whatever you do not think today becomes a part of the mandate which you have to take for surviving tomorrow. Like this is a simple thing. And maybe that is what R&D is you have to keep on moving with the concepts and the problems that society is facing it is.

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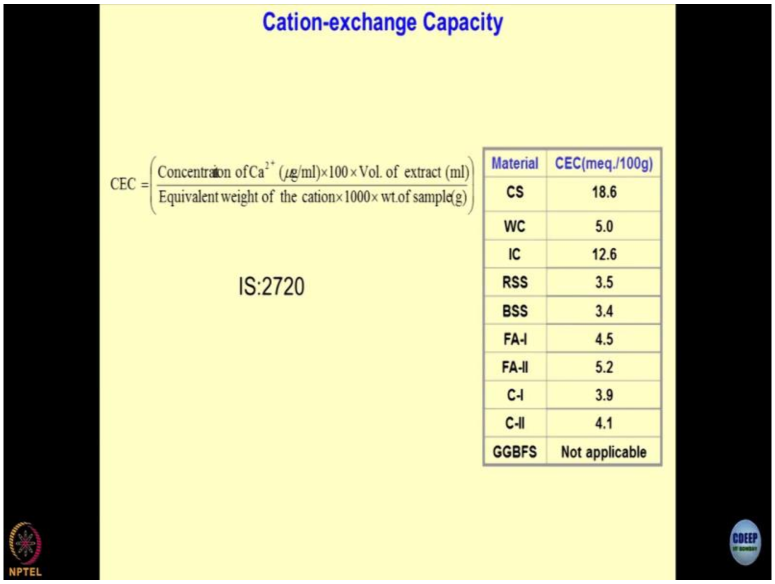

Cation-exchange Capacity

$$CEC = \left(\frac{\text{Concentration of } Ca^{2+} (\mu g/ml) \times 100 \times \text{Vol. of extract (ml)}}{\text{Equivalent weight of the cation} \times 1000 \times \text{wt.of sample(g)}} \right)$$

IS:2720

Material	CEC(meq./100g)
CS	18.6
WC	5.0
IC	12.6
RSS	3.5
BSS	3.4
FA-I	4.5
FA-II	5.2
C-I	3.9
C-II	4.1
GGBFS	Not applicable

This is a cation-exchange capacity, which you are talking about. So, and one of the ways to characterize wise would be the best way would be its CEC. So, if you see our papers which we have written, we have created a soil classification system based on SSA and CEC, and this is a published work by Shusha Lakshmi I think Dr Shrinivas Kadali. So for us, if cation-exchange capacity is known, nothing else is required. Why? Because calculations capacity includes in it the physical properties attributes, the chemical attributes.

And mineralogical attributes and all sorts of other attributes which otherwise we will not be able to detect. So I can say that cation-exchange capacity happens to be a parameter which is the holistic representation of the material. Now, the question is, how would you obtain this. So, there is a code IS code 2720 which describes this method. As I said, this is a base exchange process to take the soil allows interaction with a calcium solution, mostly calcium chloride of certain concentration.

So, when you are allowing interaction between soil and calcium chloride, all the calcium ions will get added onto the soil particle they will replace sodium ions you agree because the valence of the calcium is 2 and sodium is only 1, so higher relevancy replaces a lesser valence. So, this is the first cycle, then what you have to do is, after this interaction is over you wash the soil sample with sodium acetate what you are trying to do now you are trying to see whether sodium iron can displace calcium irons which have got adhered on the soil particles or not.

So this process has to be done several times; it is an intricate process. So, when you do this process, at the end of it, if you can measure the concentration of calcium ions equivalent weight of the cations weight of the sample and what is the volume of the solution which you have taken it is a simple titration this gives you the cation-exchange capacity is the answer to your question clear. You please read the published papers by anybody from my lab because, for us, this is the starting point.

So we do not talk in terms of size of the particle and its mineralogy separately, what we do is, we define the potential of the material to interact with the environment based on its cation-exchange capacity. And just as a quick match of numbers, what you will observe here is I have given sort of an application of how corrections capacity can be utilized to decipher the characteristic of the material.

First of all, you should realize that the units of CEC are meq/100g as per standard. What you will observe here is that cenospheres were exhibiting extremely less value of cation-exchange capacity yesterday we discussed their cenospheres quartz balls. So quartz is the least reactivity fly ashes by virtue of having some chemical adulteration in them might show you a bit high cation-exchange capacity, but the maximum cation exchange capacity comes in the soils the CS is a normal silty soil where you get very high value of cation exchange capacity and chalk where also you have the high cation-exchange capacity. So, these are tentative numbers. But these numbers tell you how reactive the system could be some time back; I was talking to you about spillage of activity, the nuclear activity of the cause of the disaster. And that spillage took place on in the water body. And from the water body, it came on the beaches, and the sands and the soil got completely contaminated. So, at that time, some people contacted me that can I create a material of a certain CEC value.

So, imagine the similarity between the cement which are being sold in the market, you have a great of the cement, is it not? Now people are asking that can you create the soil of a certain grade do you realize what is happening in the international market. So you might be very lucky to have a certain mineral in your country, but that mineral might not be available everywhere.

So, can you convert or can you alter the existing minerals to a level which becomes a medicine or it becomes a commodity for the industry?

So, this is where a lot of processes are being done and a SEMs which I showed you in the previous lecture, we are on the quartz balls you are having some deposition either because of the zeolitization or because of agglomeration of the particles which I showed you when you do dual gas conditioning, flue gas conditioning. Ultimately what happens is the CEC gets enhanced. So, this becomes a precious catalyst for me.

And these all catalysts are created by human beings. I hope you have understood the whole thing. **"Professor - student conversation starts"** Yes. Sir, Silica fumes. Their specific surface area was very high. So, it was used to be it should be highly reactive. So that cation exchange capacity of silica fumes should be high. So, you must realize that I have repeatedly been saying that what happens is because these are all gravimetric processes. **"Professor - student conversation ends"** So if you are adding material to liquid, where it floats in it you have defined the concept of titration or concept of dry gravimetric analysis.

So, unfortunately, silica fumes cation-exchange capacity cannot be obtained by this method, because the moment you make a solution out of it, they will only think like number one. So, if you have to find out the cation-exchange capacity of silica fumes, then you have to use some other method, and that would be SSA based. So, if you see the papers which are written by Susha Lakshmi and Srinivas Kadali were we have come out with generalized relationship between different parameters which are used in conventional geomechanics.

And we have related all these parameters with SSA, and CEC input in liquid plastic limit shrink a limit and specific gravity also. So, my idea was to get rid of the conventional scheme of classification, which is partial, which is not holistic, it takes into account only physical attributes and replaces the entire thing with something which is more holistic. So, this is what the state of affairs is right. One more question which might be linked with this discussion is supposed, there is a microbial activity which is harping in the soil mass or the geomaterials what will happen to the cation-exchange capacity? So, in our opinion, any process which occurs in the geomaterials can be monitored by regular sampling of the material and checking cation-exchange capacity. So, if CEC changes that change because of some mechanism and then I can trace back that is because of what. So, this is a present and future of the subject I hope you got enough ideas

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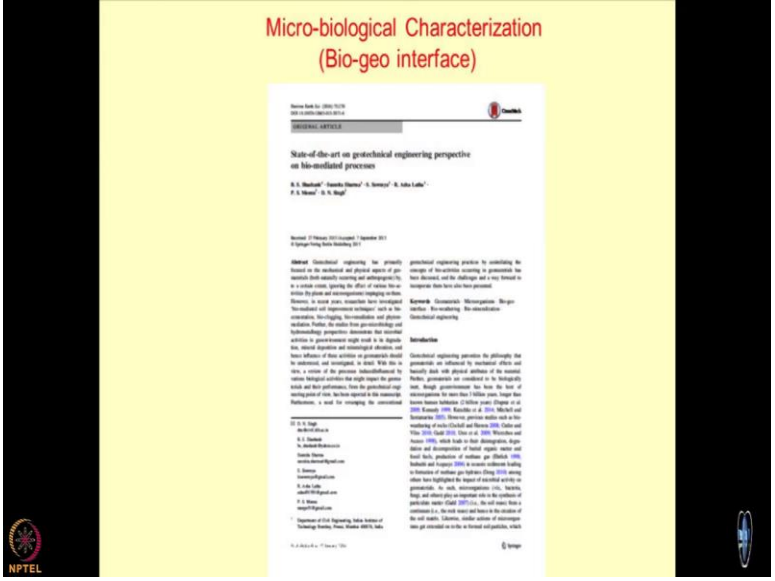

Micro-biological Characterization
(Bio-geo interface)

ORIGINAL ARTICLE

State-of-the-art on geotechnical engineering perspective on bio-mediated processes

Just to touch upon the microbial characterization. Read the papers by Shashank and Dr Sowmiya, Meenu, Asha these are my students who are working in this area, and we are probably very good papers on the microbial characterization of geomaterials from where the subject on bio-geo interface started this is a paper which referring to state of the art on geotechnical engineering perspective on bio mediated processes.

If you read the introduction of this paper, you will realize that we are absolutely anti terzhaghi geomechanics and why we are saying that all the concepts should be dry-cleaned. So, you have to read the paper to understand that twice as a type of tones, we have acquired in the process of doing R and D.

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Soil is not a lifeless entity...!

And this is the slide which I have taken from Shashank's thesis if you remember, this is the microbial activity which is harping in the Soils and which cannot be ignored different type of bacterial growth, microbiological growth and this is the harping of the biological activity on sands. So, we were talking about biosorption, biosuction, bios CEC, and so on this work is still ongoing. But very interestingly this concept started with one of my consulting projects where nobody was able to define the cause of failure of a huge raft, piled raft system.

And this piled raft system had been under severe distresses and the company was unable to start up their process. So, mechanistically everything was absolutely all light, but even then, the settlements are occurring. So that is a time when these thoughts came to our mind that let us take samples from as deep as 40,50 meter, the pipes are resting and do some microbial analysis examination, and then we realized that there was a big microbial effect on the stability of the soil.

This is all this subject started. But did you find any evidence regarding that raft settlement before yes, so, we ultimately linked everything to the microbial activities? So, the whole dimension of the project got changed, then adequate precautions were taken to not let microbial activity survive at that particular depth. From this point onward, the decay of the material also became a major thought in our research process.