

Environmental Geotechnics
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Lecture - 27
Geomaterial characterization – 7

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Slide 1

Lecture Name:
Geomaterial Characterization

Sub-topics

- **Chemical characterization**
pH, TDS, EC, BOD, COD
Sulphite and Chloride contents
Cation-Exchange Capacity
Pore-solution sampling
Corrosion potential
Sorption-Desorption
- **Thermal Characterization**
- **Electrical Characterization**

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Again, I continue with the Geometrical characterization with subtopics as chemical characterization though we have discussed how to determine pH, TDS that is Total Dissolved Solids, electrical conductivity of the soil solutions, BOD and COD, sulfite and chloride contents, then cation exchange capacity followed by pore solution sampling.

So, most of the lecture today's lecture will be discussed will I will try to cover most of the techniques used for pore solution sampling followed by most probably in the next lecture, or if possible today I will be talking about corrosion potential of soils, sorption, desorption continue with that followed by thermal characterization and electrical characterization, this is just to remind you that the characterization is still going on and we are still stuck up with chemical characterization part.

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pH determination




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

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Now, to start with pH determination this is the unit which is known as water quality analyzer and this water quality analyzer unit is connected to some electrodes. These electrodes are basically glass calomel electrodes you must have used them also earlier for determining pH of the solution of soils.

The only thing is that you have to prepare soil solutions with different liquid to solid ratios which is also known as L by S. So, depending upon different L by S pH values may change may not change; truly speaking pH value should remain same for different L by S. But it so happens if you keep on increasing more and more liquid into the solution pH may also alter because of more opportunity which oxides may get to react with water and sometimes when you are working with oxides and the materials which are amphoteric in nature which have dual you know behavior when they come in contact with water, pH may definitely change.

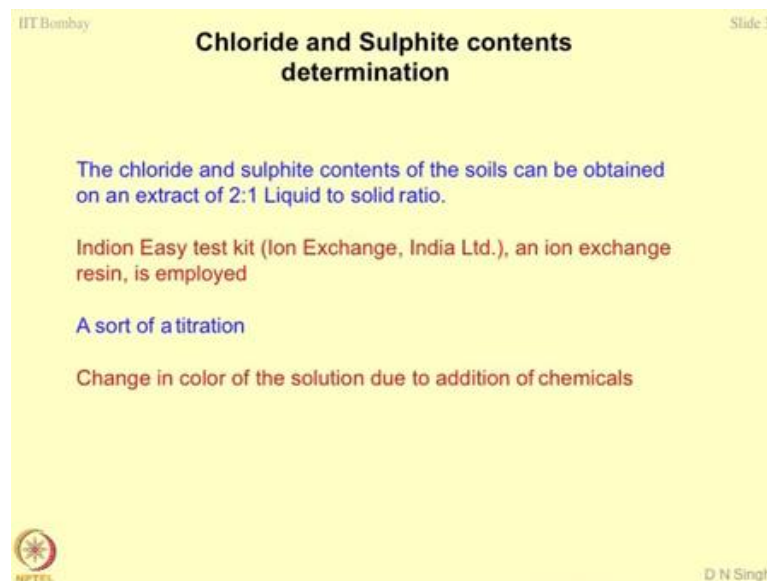
Now, this instrument is a very good tool to determine pH of the soil solution essentially what you have to do is you have to prepare a soil solution corresponding to a liquid solid ratio and then filter out the liquid part and that liquid part is normally kept in this beaker, the glass calomel electrode is dipped into this and then you can read out reading directly.

However, you have to be very careful that pH normally gets influence by the temperature. So, this instrument also records temperature simultaneously and normally at room temperature that is 25°C the pH values are reported. So, you can apply the

correction depending upon the values which you were obtaining if the temperature is not same as 25°C. You can use this water quality analyzer also for determining total dissolved solids which is an indicative of turbidity of the solutions, if you cannot use a UV spectrophotometer, then this is also a good technique.

You can determine electrical conductivity of the solution of the soil corresponding to different L/S ratio and then ultimately electrical conductivity can also be linked with TDS of the suspension in the material, you can find out chemical oxygen demand as well as biological oxygen demand. And based on these parameters you can again classify the soil and later on we will notice that these parameters are very handy in defining the corrosion potential of the soil mass. So, particularly pH and electrical conductivity and sometimes another word which is used as redox potential, I will be talking about that later on. So, this is simple device to determine all these parameters and different liquid to solid ratios.

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Chloride and Sulphite contents determination

The chloride and sulphite contents of the soils can be obtained on an extract of 2:1 Liquid to solid ratio.

Indion Easy test kit (Ion Exchange, India Ltd.), an ion exchange resin, is employed

A sort of a titration

Change in color of the solution due to addition of chemicals

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Now, most of the time when you are working as a geotechnical engineer for site investigation, your profession demands that you should understand what is the concentration of chloride and sulfide ions in the soil as well as in the ground water. Can you guess where this type of studies would you more useful or applicable.

Student: Nearby the sea shore.

That is right particularly near by the sea shores how to estimate in very gross terms what is the degree of contamination of the soil mass of the groundwater. Particularly in Bombay region when you are doing piling activities it is mandatory to understand what type of soils and what type of ground water conductivity and chloride and sulfide concentrations are available and then what will happen suppose if I determine that the concentration is very high. So, what you should do then as a protective measure? Then you should use some sulphate resistance cements.

So, this is where the sulphate resistant cement becomes mandatory then you can also coat the piles by using some polymers or the resins or some epoxies. So, the normal practice is the pile should be protected against any corrosion due to water or soil which is having very high concentration of chloride and sulphates. So, here the normal procedure is that you take soil sample, dissolve it in water and make a liquid solid ratio of 2 is to 1 that is L by S equal to 2. And we go for indirect titration and that titration is done with the help of ion exchange units which are known as Indion easy test kits, these test kits are very useful basically this is ion exchange resin and if you drop it in the solution of soil mass or soil solution their color changes.


So, depending upon the number of drops which you have added to the solution of a particular resin you can find out roughly what is the concentration of chloride and sulfide ions in the soil mass. So, it is a sort of a titration technique. Very helpful for people who are working in field and those who want to estimate the what amount of chlorides and sulphides are present.

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IIT Bombay **Determination of Cation exchange capacity (CEC)** Slide 4

Is the amount of cations a soil can hold.
Summation of exchangeable cations (Na^+ , K^+ , Ca^{+2} and Fe^{+3})
Factors affecting CEC are: charge carrying capacity of the soil, pH, ionic strength of the pore-solution and presence of salts.
Guidelines presented by IS 2720 (part XXIV, 1976) and (EPA SW-846) are followed for the determination of CEC of the soil sample.

IS 2720 (Part XXIV 1976):
The sample is first treated with hydrogen peroxide (H_2O_2), and boiled thoroughly for 1 h to remove organic contents.
The treated sample is oven-dried and its 5 g is mixed with 50 ml 1N Sodium acetate (CH_3COONa) solution with pH=5.
This mixture is digested in a boiling water bath for 30 min., with intermittent stirring, and later centrifuged at a speed of 5000 to 6000 rpm, for 15 min.
The supernatant liquid is discarded and the sample, settled at the bottom of the centrifuge tube is again treated with 50 ml of 1N CH_3COONa solution (pH=5) and centrifuged. Repeat this process thrice, so as to ensure exchange of Ca^{2+} in the soil by Na^+ , completely.

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And this can also help you in determining what is the corrosion potential of the soil. Now, in my previous lecture I have talked about the cation exchange capacity of the soil mass, but I did not talk about the whole methodology of determination of CEC. But you have noticed that CEC is a parameter on which lot of things depend and for the entire property of the soil mass depends.

So, with this in view I thought of covering the steps which are normally adopted for determining CEC of the soil mass. Basically, what is CEC? The classical definition is that this is the holding capacity of the soil of any contaminants or cations so; that means, this is an amount of cations a soil can hold easily. Now, this is where I was using the term parking of the cations on the soil grains all right and all of you are aware by this time that CEC plays a very important role in soil contaminant interaction.

You can also put it as that this is the summation of exchangeable ions that is Na^+ , K^+ , Ca^+ , Fe^{+3} and Fe^{+2+} on the soil grains. So, factors affecting CEC are basically the charge carrying capacity of the soil. Now my question to you is how would you determine the charge carrying capacity of the soil? We always assume that clays are negatively charged particles, but then the question is how would you estimate, that what is the amount of the charge which is available on the grains of the soil. Is there any method which comes to your mind?

There is a method known as zeta potential determination. So, essentially zeta potential describes how much charge is available on the surface of grains of the soil. Now, if you go in these types of studies you can very easily answer the questions that why clays cannot be compacted beyond a certain limit. So, apart from a double layer formation because of the negatively charged particles you cannot put two particles together just because of the mechanical energy.

So, there is a limitation of the compaction process, but if you remember if you go for some energy principle and if you expose the soil mass to some other energy field particularly electric or electromagnetic then what happens to this double layer of water surrounding the grains of the soil gets reduced and soils can be compacted very easily. So, based on this technique the genesis of electro kinetic remediation takes place or the consolidation which is accelerated due to electric field application.

So, this is important factor that CEC gets influenced by the charge carrying capacity of the soil, I think you can notice this point that as the clay particles are negatively charged when they come in contact with the cations there is a sort of a neutralization of the charge on the surface clear. So, this is the bond which is formed at the time of soil-contaminant interaction and rather than contaminant if you are dealing only with water then there is a dipole formation or a double layer formation of water, CEC will also depend upon the pH of the solution.

So, on both sides of the pH equal to 7 that is acidic and basic environment the soil exhibits active behaviour. So, this part you may come across later on when I will talk about the influence of pH on the corrosion potentials of the soils. Ionic strength of the pore solution, the type of cations the type of ions which are present in the pore solution will also influence the cation exchange capacity of the soil and of course, the presence of the salts.

Now, there are two codes normally which are followed, one is IS 2720 and the EPA SW 846 which are followed to determine the CEC of the soil sample. Normally we follow IS 2720. So, the sample is first treated with the H_2O_2 . And what it does? If you treat the sample with H_2O_2 it will remove the organic contents and boiled thoroughly for 1 hour. So, this way you can get rid of all the organic contents which are present in the soil mass, you should notice here that why you want to do this type of treatment to the soil.

If organic content is too much the reactivity of the soil will be more or less it will be less, that is one of the reasons why you cannot stabilize organic and marine soils, it is very difficult to stabilize them you got the point. So, organic soils cannot be stabilized so easily first of all they themselves not stable, second is their water holding capacity is very high. So, even if you put the cement in then what is going to happen? Cement will not get any water out of the soil to get cured and to form a bond because these soils will not release water so easily.

So, this type of mechanism goes on. So, there is the reason that you have to remove organic contents from the soil to determine its real potential. So, the treated sample is oven dried with 5g and it is mixed with 50 mL of 1N CH_3COONa solution with pH 5. Now this mixture is digested in a boiling water bath for 30 min with intermittent stirring and later centrifuge at a speed of 5000 to 6000 RPM for 15 min.

The supernatant fluid or liquid is discarded and the samples settled at the bottom of the centrifuge tube is again treated with 50 mL of 1N CH_3COONa . CH_3COOH is acetic acid. So, CH_3COONa is sodium acetate solution again with pH 5 and centrifuged repeat this process thrice so as to ensure exchange of calcium ions in the soil by sodium ions. So, this is the first step where you are replacing all the sodium ions with sorry calcium ions with sodium ions why because you are using sodium acetate solution.

So, is the leaching of calcium ions out of the sample of the soil all those calcium ions gets replaced by sodium ions what we will have to do next is we will have to replace the sodium ions which are getting fused on the soil by treating with some other chemical.

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This sample is treated with 1N Calcium chloride (CaCl_2) solution and is again digested and centrifuged. This process is repeated thrice, so as to ensure exchange of Na^+ by Ca^{2+} .

The sample is treated again with 50 ml 1N CH_3COONa solution (pH=7) and again digested and centrifuged. This operation is performed thrice.

The resulting supernatant from the last three steps is collected in a 250 ml volumetric flask, and the concentration of Ca^{2+} present in the solution is determined using the Atomic Absorption Spectrometer, AAS.

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

Minerals Present in soils(XRD)	CEC (meq./100g)
Montmorillonite	18.6
Kaolinite, Illite	4.989

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So, this is the first step of the process. Now, this sample is treated with one normal calcium chloride solution. So, this is where sodium is being replaced by calcium ions and it is again digested and centrifuged this process is repeated thrice. So, has to ensure exchange of sodium ions with calcium ions, Sangeetha is realizing that it is a very tough task [Laughter] most of the things require lot of patience as I say again and again. Now, this sample is again washed with sodium acetate. Why you are doing this now? You are trying to check whether further sodium can replace calcium or not, if not your process is done. But if there is a change your process is not done you have to repeat the process again.

So, this sample is treated again with 50 mL of 1N CH_3COONa solution of pH 7 and again digested in centrifuge this operation is performed thrice. The resulting supernatant from the last three steps is collected in 250 mL volumetric flask and the concentration of calcium and present in the solution is determined using AAS that is a atomic absorption spectrophotometer.

Now once you have done this you can obtain CEC.

$\text{CEC (meq. /100g)} = (\text{Concentration of Ca}^{+2} \text{ or Na}^{+2} \text{ or K}^+ (\mu\text{g/mL}) \times 100 \times \text{Volume of te extract (mL)} \times (\text{L/S ratio})) / (\text{Eq. Weight of the cation} \times 1000 \times \text{wt. of the soil (g)}).$

Just to give you some idea about what are the CECs of different minerals and hope you will appreciate why some minerals are very active and why some minerals are not active this table gives you a clear picture, if you find out the minerals by using XRD X-ray Diffraction technique for montmorillonite the CEC is 18.6. And for kaolinite and illite CEC comes out to be approximately 5. So, almost it is 3 to 4 times difference between the cation exchange capacity of the clays itself alright.

So, because kaolinite and illite itself is a clay and montmorillonite is also a clay mineral which is one of the reasons why water absorption capacity of montmorillonite is much more higher than kaolinite. But in classical geomechanics we always say in that what type of bonds are available in montmorillonite, it is a Van der Waals forces, is it not. So, because of Van der Waal forces the water absorption capacity is more, that is where we have not talked about in the chemical potential of the soil or chemical aspects of the material.

So, by putting cation exchange capacity also in conjunction with the platelet theory you can find out what would be the total potential of the soil towards its activity or reactivity with contaminants or the environment is this clear.

Now, this CEC value can be utilized for further classification of the soil. So, this seems to be very good tool, if you want to differentiate between coarse-grained soils and fine-grained soils and within fine-grained soils also you think or you imagine a situation where each mineral is specified with a CEC value. So, the moment you get the CEC value you need not to go for even XRD analysis which again is a very costly and expensive procedure.

So, what you can do is you can ascribe this number and you can say that this soil has more predominance of montmorillonite and so on. So, this again seems to be a good classification scheme based on chemical composition of the soil mass.