Characterization of Construction Materials
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Lecture 2
Characterization of Construction Materials An Introduction Part 2

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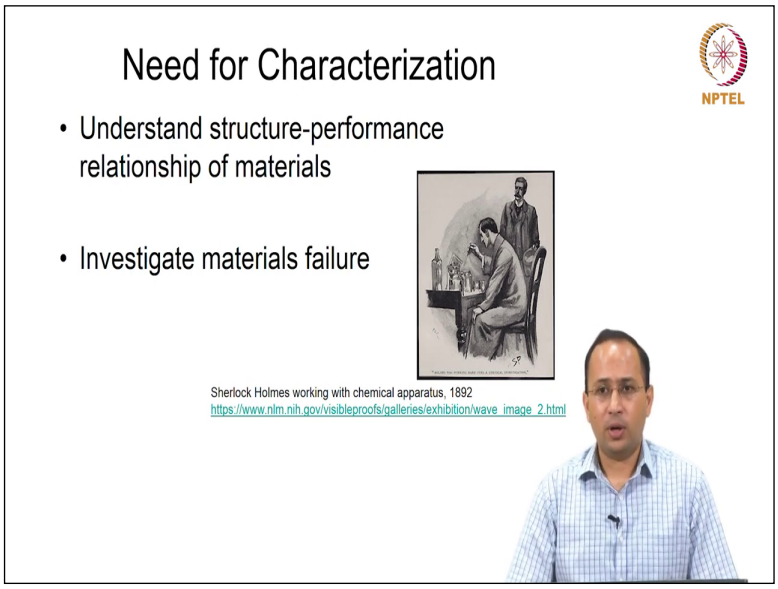

Hello everyone, this is Piyush Chaunsali and I will be co-teaching this course with Professor Manu Santhanam. What I will do in this second part of the introduction lecture, is to further motivate you to understand the need for characterization. So, basically characterization can help us understand better structure performance relationship of materials. One simplest example could be difference between diamond and graphite.

Both are forms of carbon, but exhibit very different properties. So what makes on form of carbon show varied properties than the other? So what we are targeting is structure-performance relationship. What are the features in structure of materials that influence the performance? So that will be the overall theme of this course. We will try to better understand this relationship.

Also this understanding of structure performance relationship is helpful to investigate material failure. Why materials fail? Obviously, it depends on some characteristics materials exhibit. What we are talking about is structure and also how the structure changes when the materials are exposed to different environments. So, basically by understanding the structure-

performance relationship we can better understand the material's failure. So, these will be two broad objectives of this course.

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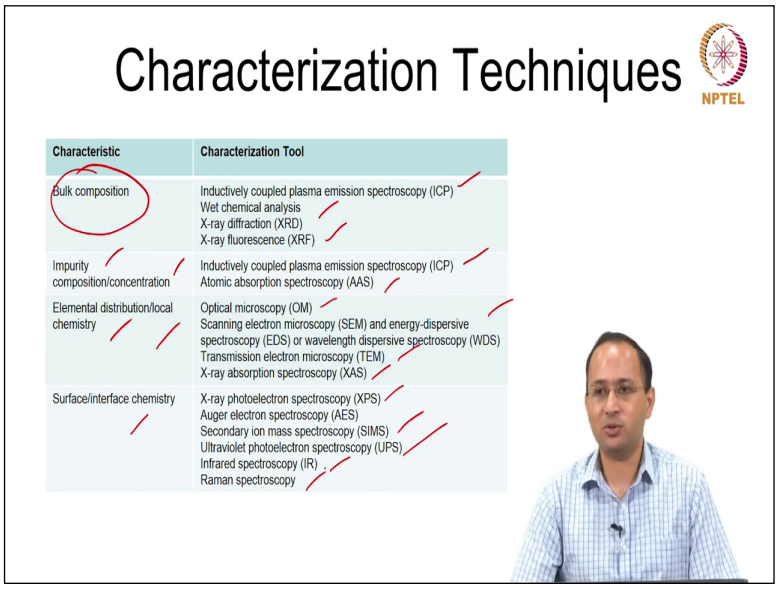

So, there are lot of characterization techniques which we can use to better understand this relationship. The kind of tool we need depends on the characteristic we are aiming at. For example, to determine the bulk composition of material then we can use these techniques:

1. Inductively coupled plasma emission spectroscopy ICP
2. Wet chemical analysis
3. X-ray diffraction
4. X-ray fluorescence.

Similarly, to determine impurity composition and concentration we can use ICP and Atomic absorption spectroscopy (AAS).

Elemental distribution and local chemistry can be determined using Optical Microscopy, Scanning Electron Microscopy, Transmission Electron Microscopy and X-ray Absorption Spectroscopy. We will cover a few of these techniques in this course.

Surface and interface chemistry can be understood by X-ray Photoelectron Spectroscopy (XPS), Auger Electron Spectroscopy, Secondary Ion Mass Spectroscopy, Ultraviolet photoelectron spectroscopy, Infrared spectroscopy, Raman spectroscopy. So, there are lot of spectroscopic techniques which we can use to determine the surface and interface chemistry.

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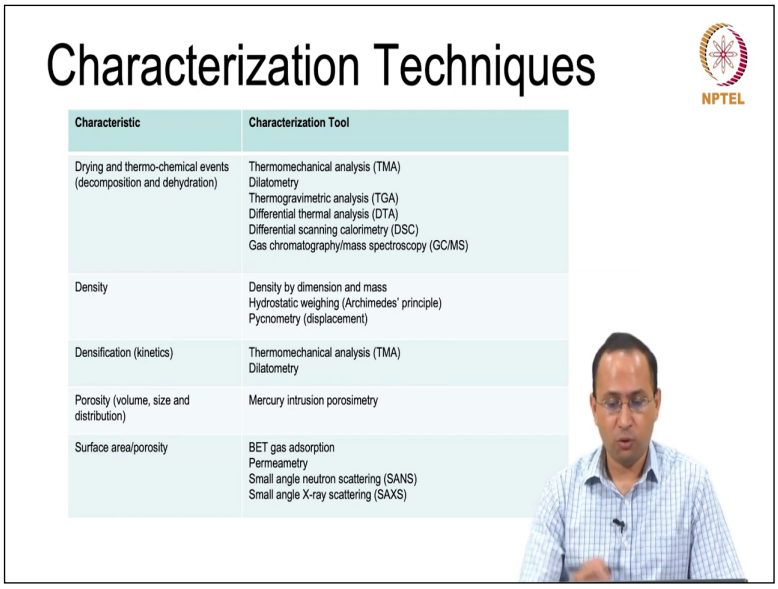

If you are looking at drying and thermo-chemical events like decomposition and dehydration, we can use Thermomechanical Analysis (TMA), Dilatometry, Thermogravimetric analysis (TGA), Differential Thermal Analysis, Differential scanning, Calorimetry, Gas Chromatography, Mass spectroscopy. So, these are the techniques which we can use for drying and thermo-chemical events. Similarly the technique, the characteristics and corresponding characterization tools which are available have been listed.

So you will be covering, few of these techniques. Similarly, if you look at the surface area, we'll be covering BET gas adsorption in the course. But you can also use permit Permeametry, Small angle neutron scattering (SANS), Small angle X-ray scattering (SAXS). So, these are the tools we have at our disposal. Similarly there are a bunch of tools to determine density, also particle/grain size, distribution, morphology, and texture.

Phase identification, thermal events like phase transition, transformation can be captured using Differential thermal analysis (DTA), Differential scanning calorimetry (DSC), Thermomechanical analysis (TMA). So, depending on the characteristic we are looking at, we can pick the characterization tools. We cannot use all the tools for all the characteristics. If we have specific characteristics then, we pick the suitable characterization tools which can give us some information.

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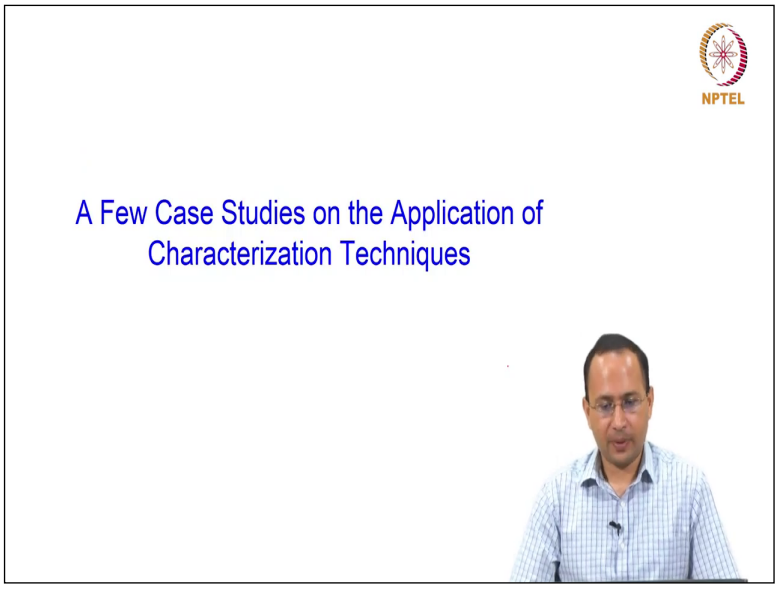

The rest of this section, I will be discussing a few case studies I have picked from various, not just limited to construction materials, but from different materials, just to motivate you to go for characterization.

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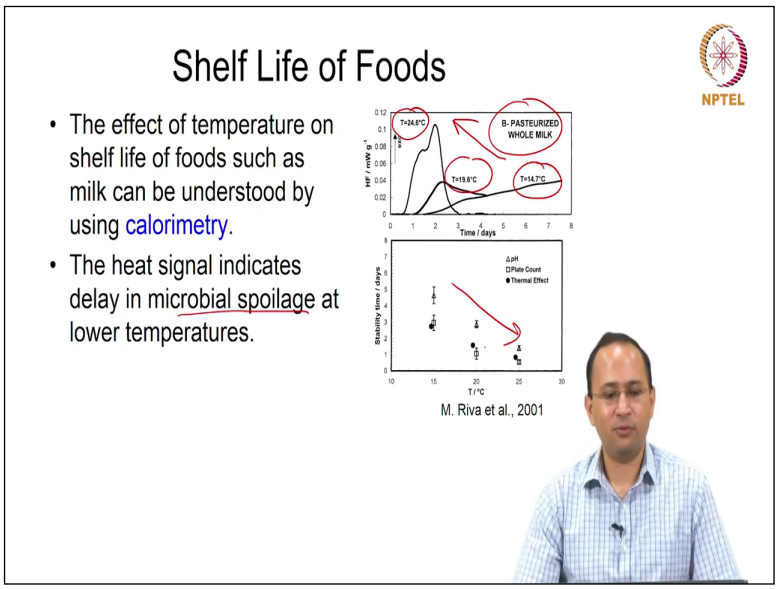

So, let us start with Calorimetry. Although we will look at the application of calorimetry in construction materials, you can see here calorimetry can be used to even monitor the shelf-life of foods. So in this example, we are looking at the effect of three different temperatures and how that affects the heat evolution.

In this case, we are talking about microbial spoilage. So, with the increase in temperature, we accelerate the microbial activities that correspond to this increase in heat, that affects the shelf-

life. So, as you see here, the shelf life decreases as temperature increases. So, as long as there is a heat signal, we can use calorimetry to monitor that process. In this case, we see it is applicable to determine shelf-life of foods.

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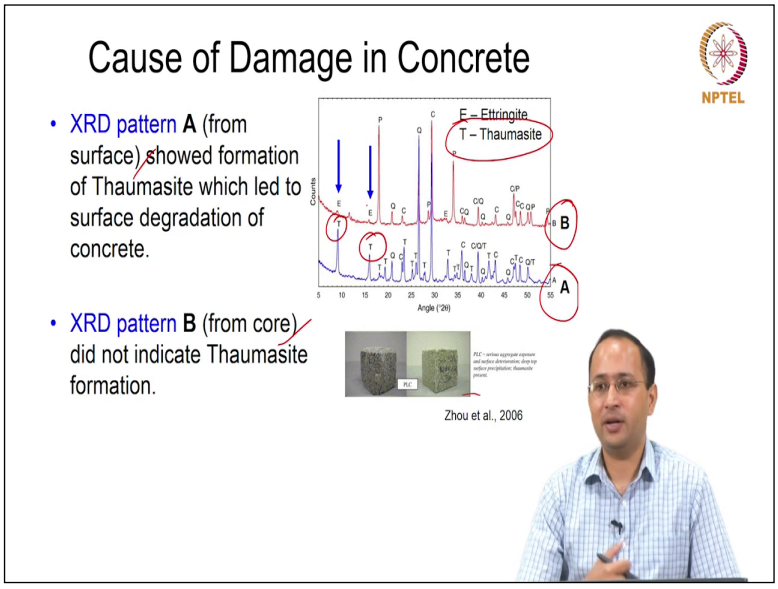

X-ray diffraction is commonly used technique in construction materials and it can be used to determine or identify the cause of damage in concrete. In this example, what we see in this plot, two x-ray diffraction patterns of samples - one taken from core and one from surface. Obviously one from core did not have any damage; but one with the surface had the damage.

They wanted to know what the cause of damage was. So, in XRD pattern A, we see presence of thaumasite - which is one of the phases; but in pattern B we do not see thaumasite and only see ettringite. So, they attributed the presence of this thaumasite phase to the damage of concrete. So, XRD is a tool which can be used to identify crystalline phases in materials. So in this case, it could be used to identify the damage of concrete.

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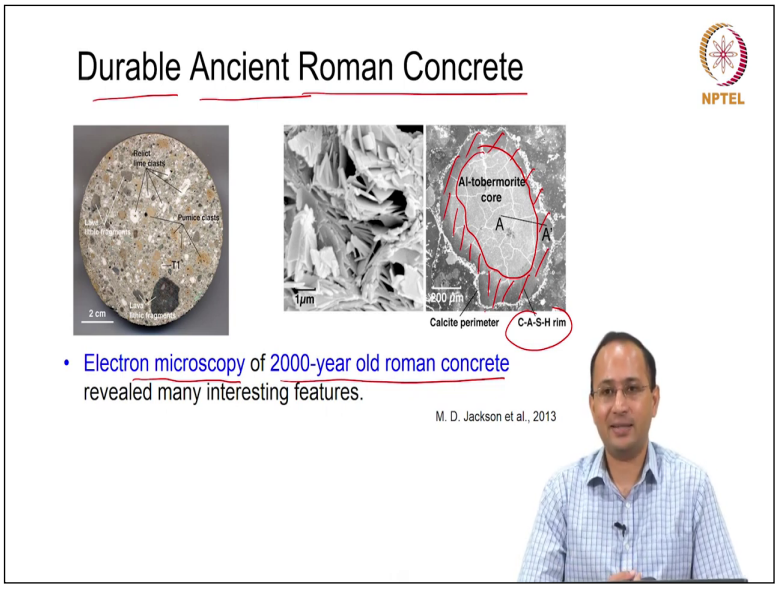

So far we talked about the damage, but sometimes, it is also important to understand why materials last long? This is an example of Roman concrete - Ancient Roman concrete, which was in good condition. It was 2000-year old Roman concrete buried in seawater and researchers wanted to understand what is in this concrete which made this last so long. So, we are talking about durable Ancient Roman concrete.

So, electron microscopy is one of the tools which can give us insight about the microstructure. So, it can tell us what kinds of phases are formed in material. So, here they found the presence of Al - Aluminium Tobermorite in the Roman concrete. Here, we can see different phases. In the core we have Al-Tobermorite core surrounded by C-A-S-H rim - Calcium aluminosilicate hydrate, which we usually see when we have OPC with supplementary cementitious materials. So, these kinds of insights can be gained by using Electron Microscopy.

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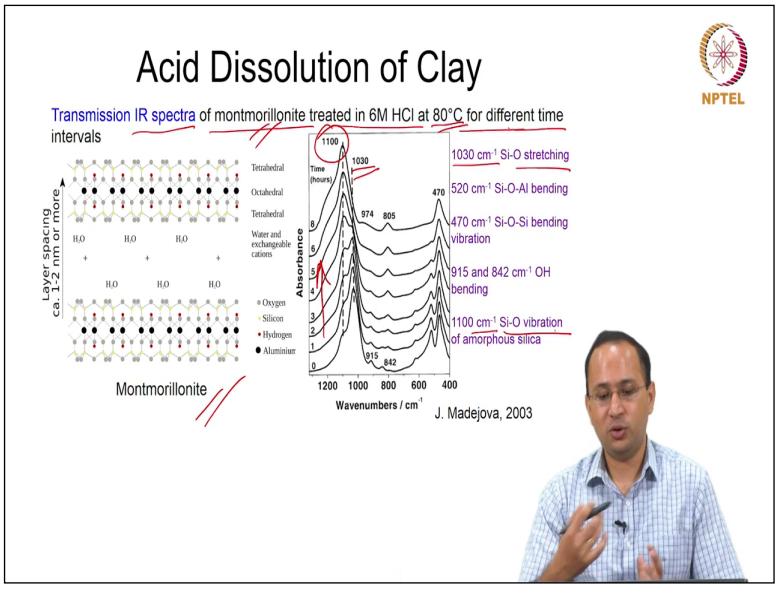

One of the techniques which will be covered in the class will be Infrared Spectroscopy. So, in this case, the transmission IR spectra can be analyzed and used to monitor the changes in montmorillonite clay (a type of clay), when it is exposed to 6M hydrochloric acid at 80°C, for different time intervals. So, you have the structure of montmorinollite in the figure.

How the structure changes - basically the spectroscopy can be used to understand the bonding environments. See here, we get these peaks. For an example, the peak at 1030 wave number tells us about the SiO stretching. How this peak is changing as we are exposing clay to this 6M hydrochloric acid at 80 degree Celsius. And here you see for 1-8 hours. So what you see are some changes in this 1030.

The peak at 1030 starts disappearing and you finally see 1100 cm^{-1} peak which corresponds to Si-O vibration of amorphous silica. So, these kinds of changes in the bonding environments can be captured using infrared spectroscopy.

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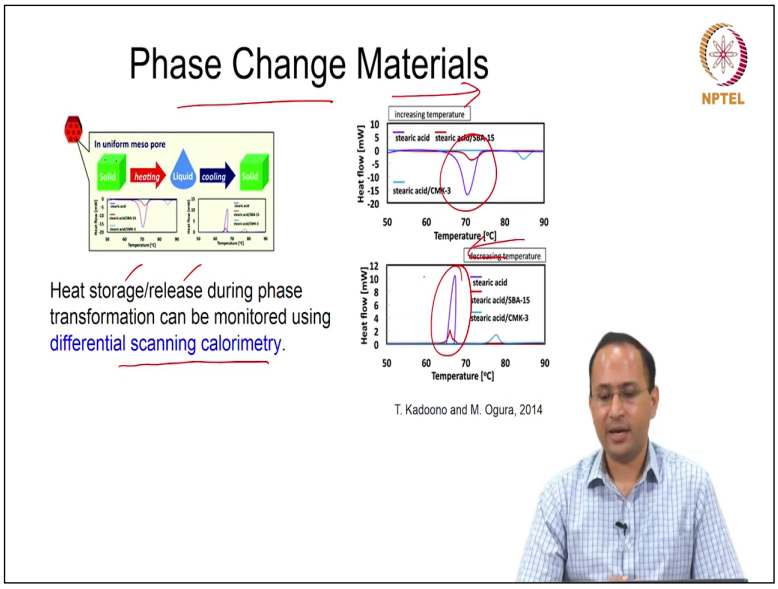

We earlier talked about Heat Signal. We can also quantify how much heat is absorbed and how much heat is released. So, basically heat storage and release during phase transformation can also be captured using the Differential Scanning Calorimetry. So, you might have heard of phase change materials.

Phase changing materials – We have a solid, exposed to temperature rise. When the melting starts you have a liquid and that absorbs lot of heat. Then, upon cooling that will transform to solid that will release heat. So, these kinds of changes can be captured using Differential scanning calorimetry. So, here in the plot what you see, as the temperature is increased you see endothermic peak, because the melting takes place.

And when you are decreasing the temperature, in this case you see exothermic peak. So, these kinds of change can be captured using Differential scanning calorimetry.

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So the idea is to have a multi-pronged approach. So, it is very important to use multiple techniques to corroborate findings of a particular technique. Lot of times, we may get something by using one technique. But it may not give us all the information. So, sometimes we need a validation. So, it is always good to keep in mind that you need to use multiple techniques which will corroborate our findings because each technique has its limitation.

For an example, X-Ray Diffraction is widely used to identify crystalline phases. In case your material has amorphous phase how will you identify it? So, you may have to use some other techniques. So, with the characterization tool it's important to keep in mind that you need to have a multi-pronged approach. You need to look at the problem from multiple angles. And maybe use different techniques which will be complementary.

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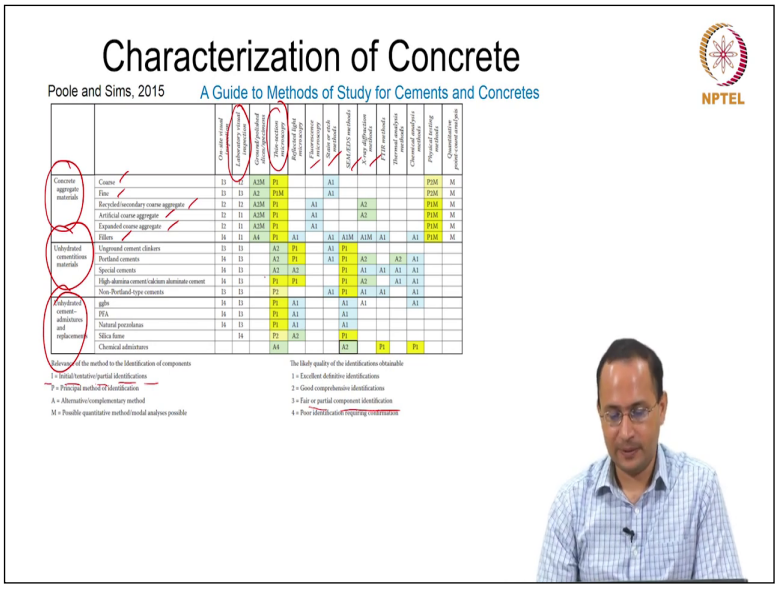

So, as far as characterization of concrete is concerned, there is a guide to methods of study for cements and concrete. What kind of methods can be used and what is the limitation. So, in this table, we have concrete aggregate materials in the first column, unhydrated cement and mixtures and replacement. If you want to study these materials, aggregates will be coarse, fine, recycled, artificial fillers.

So 'I' means Initial/tentative/partial identification. So based on the technique, on top what you see are the different techniques which we can use, on site visual inspection - first one. So, I3 means it is tentative and it provides initial assessment - fair or partial component identification - it is not excellent, you will get some idea and you will have to use some other techniques. So, if you go to laboratory visual inspection, and for thin section microscopy we see P1.

So P means it is a principal method of identification. One Excellent definitive identification so, it will tell for sure, what is in the material. So, if you go towards P that means you have more confidence. So that is what you see for different techniques. Here you have Reflected light microscopy, Fluorescence microscopy, SEM, XRD, so there are a bunch of techniques.

You can also see to understand these materials what are the limitations and what we can get. So, basically P is principal method of identification, P1 will be where you can get excellent definitive identification.

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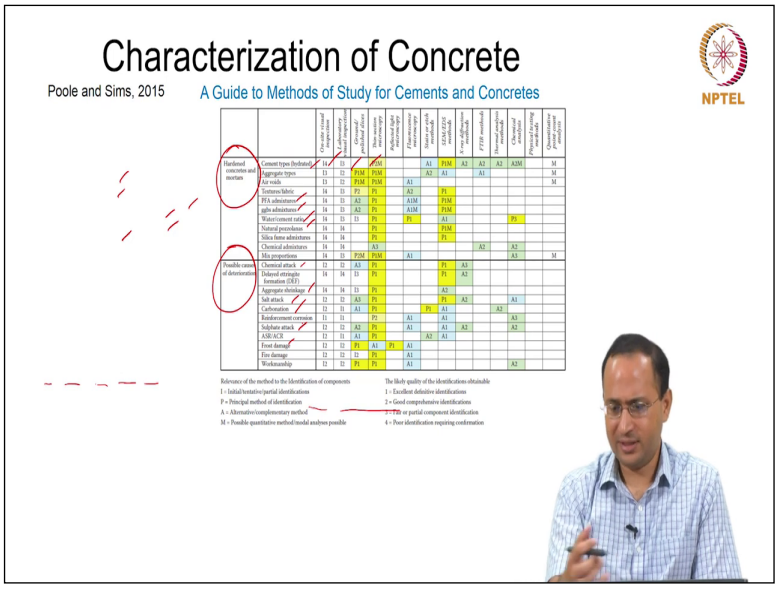

Here is another table. Now, we are looking at Hardened concrete and mortar so what type of cement, cement types, aggregate types, air void, admixtures, GGBS, water/cement ratio, pozzolans. And then, we are also sometimes interested in various type of degradation mechanisms - ASR, sulphate attack. So, this, gives us some information as to what kind of techniques, we can use for better characterization of concrete. So it can serve as a guide to various methods to study cements and concrete.

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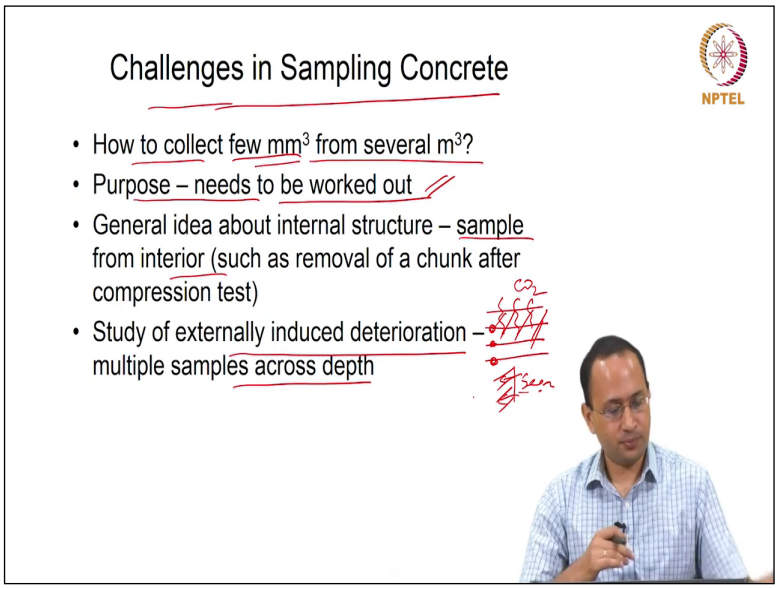

There are challenges in sampling concrete. It is not that easy because we want to get information of concrete, which is in structure. So, how to collect small amount of concrete from large? If we are talking about a structure, means we are talking about either a building, road,

bridges. So, you have concrete in that structure and because we cannot test the whole structure, we have to sample small amount, then use that to analyze.

So that is challenging. So that is what we will discuss - what are the challenges in sampling concrete. So for that, the purpose needs to be worked out. What is the purpose we want? For an example, do we want to sample to determine compressive strength of concrete or do we want to look at the bulk composition or do we want something else? So the purpose is very important, because that will influence how much amount we need.

Also we need to have a general idea about internal structure - suppose you tested a sample under compression test. Obviously it broke. And you take a small chunk to be used for other tests. Suppose, you are interested in determining porosity, so, because of that loading it may be possible that the chunk has micro cracks. So is it appropriate to do porosity test on the chunk from that broken sample from compression test? So those are the things we have to think about.

As far as deterioration is concerned, where we have external media, it is important to collect samples across the depth, because the exposure may be different. For an example, if we talk about the carbonation, you have concrete sample carbonation. So depending on the depth you will have different kind of phase assemblage. The sample taken from the surface will be different than the samples that are taken from few centimetres below surface.

Similarly, if you have a concrete exposed to sea, depending on how deep you are in concrete that affects the kind of properties you have. So these are the challenges in sampling concrete and we will talk about that in the course.

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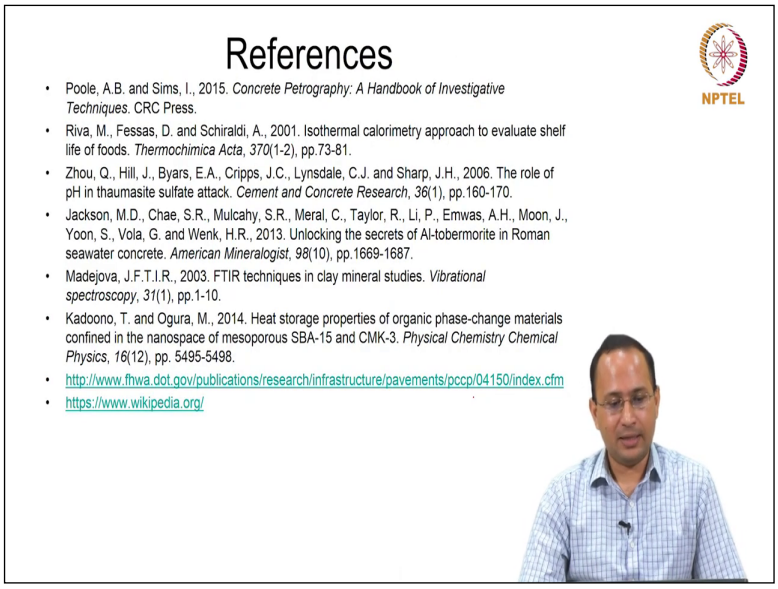

So with that I will conclude this part of the lecture and here are the main references which were used in this lecture. Thank you.