

Characterization of Construction Materials
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Lecture 3
Structure of Construction Materials an Overview Part1

Hello everybody. So welcome to the first segment of this course on characterization of construction materials before we really get in the characterization techniques. We would like to see some basic understanding of different types of construction materials. So, the ideas to provide, you overview of what you already learnt in courses before and you may have taken this courses either during your undergraduate program or postgraduate programme depending on whether you are a masters' student or Ph.D. student.


In many cases the kind of detail that you would have covered may not be 100% sufficient to really meet the demands of understanding characterization techniques as applied to these materials. But the exposure that you may have had while doing courses on design for instance could have brought into contact with the techniques are used for understanding the properties of these materials. I am just going into some basics of four primary classes of construction materials.

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Introduction


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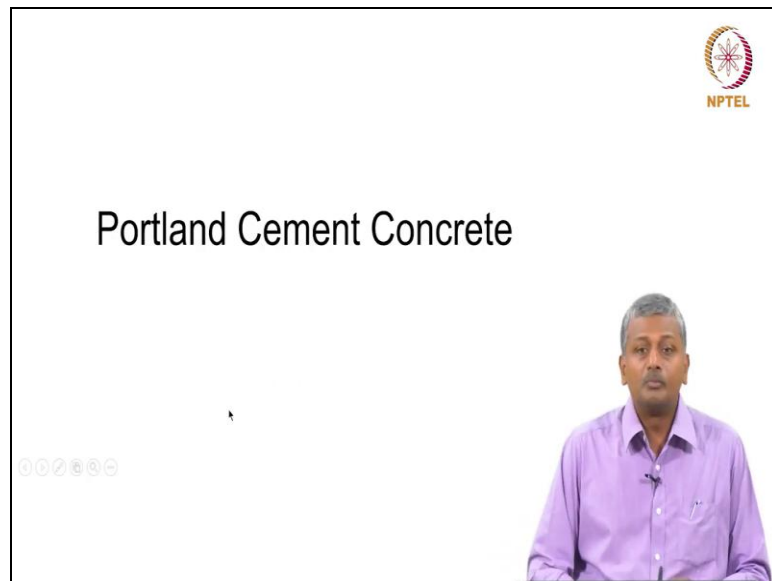
- Before proceeding to characterization techniques, we should at least have a basic idea about the structure and properties of the material
- Enables the selection of an appropriate technique of characterization!
- Let's look at: Portland cement concrete, asphalt concrete, steel, and polymers



We will mainly look at steel, concrete, Asphalt, Polymers and plastics and their importance for in understanding structural materials family. Primarily, we need to have a basic idea about the

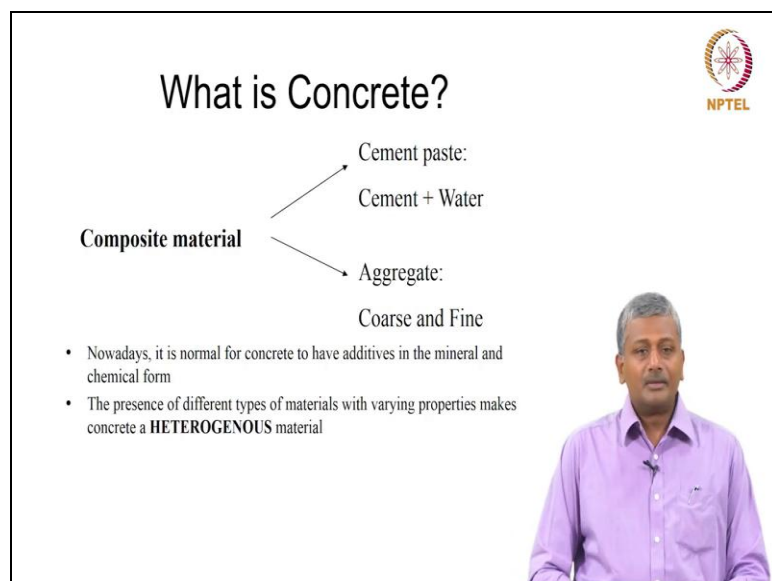
structure and properties, and then once we know this, we can actually select appropriate techniques to look at specific application of characterization techniques.

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Let us talk about Portland cement concrete first.

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And we will try and see to adopt the material science approach to understanding this material. If we try to look at Portland cement concrete, it is actually composite material composed of two different phases. The continuous phase in Portland cement concrete is cement paste phase and the discontinuous phase is the aggregate phase. What do I mean by continuous and discontinuous? Continuous means which is providing the entire volume, discontinuous means we have discrete particles of aggregate which are suspended in a continuous medium of the paste.

Now you all know very well that aggregate is composed of fine and coarse aggregate depending about the size of these materials. These days we do not just have cement, water and aggregate in concrete but also have additives basically of two types i.e. mineral additives and chemical additives. Mineral additives are the pozzolanic materials like fly ash, silica fume and hydraulic materials like slag.

We also use inert fillers, which are used to help in optimising the particle size distribution of a material. When we use all these materials in combination what we are actually looking at is different size scales. We talk about particles which may be as small as a few microns like silica fume for instance. The particle size of silica fumes is about 0.1 Micron. On the other hand, we have coarse aggregates which are 20 millimetres or sometimes even more.


We have a complex blend of the particles of different size ranges which leads to inherent heterogeneity associated with concrete. This makes it very difficult to completely design concrete to meet specific demands. But we get an approximate range of properties with some characteristic design methodologies. Heterogeneity of concrete is the challenge that we really will come across even with respect to characterization.


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Cement composition

OXIDES	COMPOUNDS*
CaO (C)	C ₃ S
SiO ₂ (S)	C ₂ S
Al ₂ O ₃ (A)	C ₃ A
Fe ₂ O ₃ (F)	C ₄ AF
MgO, SO ₃ , Na ₂ O, K ₂ O	Gypsum (CaSO ₄ .2H ₂ O)
Minor oxides	

Note: Water (H₂O) is written as H in cement chemists' notation





Coming down the basics of cement itself you all know that it is cement is composed of several different types of oxides. The primary ones are calcium oxide, silicon dioxide aluminium oxide and iron oxide. These are represented as C, S, A and F in cement chemistry notation of course it is little bit different as compared to regular chemistry because C would be carbon in regular chemistry, but here C is calcium oxide.


There are also other oxides, magnesium, sulphate Sodium and potassium oxide which are responsible for some interesting characteristics of cement especially for hydration process cement with water. There are other oxides which do not have a major bearing effect on the properties of cement except for some additional characteristics like colour and so on. But for the most part, the properties of cement with respect to its hydration with water are based on these primary oxides present in Cement.

During the process of cement manufacturing cement, it is known to be produced of an intimate blending and burning or calcination of limestone and clay which leads to the production of these compounds from the oxides. Once again by using cement chemistry notation, C_3S is nothing but tricalcium silicate i.e. $3CaO.SiO_2$ and C_2S is dicalcium silicate or $2CaO.SiO_2$.

C_3A is tricalcium aluminate or $3CaO.Al_2O_3$ and C_4AF i.e. tetra calcium aluminoferrite which is $4CaO.Al_2O_3.Fe_2O_3$. These are approximate chemical formulation for the compounds forming out of the clinkering process in cement manufacture. These are not exact chemical formulations like what we know for regular salts which we deal with reagent grade chemicals that we use in the laboratory. Those are very specific formulations, but these are compound formulations that have several different compositions, but the average composition is represented by these formulations. During the final process in manufacturing of cement, gypsum is added to work as a setting regulator. Gypsum is generally written as calcium sulphate with two molecules of water or $CSH_2.H_2O$ or water is written as H in cement chemistry notations and that is why 2 molecules of H_2O becomes H_2 .




























We write these formulations to simplify the chemical formulae otherwise the compound compositions may become too large.

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Typical properties

<p>Composition:</p> <ul style="list-style-type: none"> •C₃S: 45 – 60% •C₂S: 15 – 30% •C₃A: 6 – 12% •C₄AF: 6 – 8% •Gypsum ~ 4% 	<p>Physical properties:</p> <ul style="list-style-type: none"> •Specific gravity: 3.15 (solid); 1.5 – 1.6 (bulk powder) •Fineness: 300 – 350 m²/kg
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Typically, most cement will be very rich in tricalcium silicate or C₃S. OPC will be primarily based on C₃S. Typical ordinary Portland cement at least have greater than 50% of C₃S although the range can be from 45 to 60%. C₂S will be typically half of C₃S.

If we add silicates together, it will be around 70 to 80% of the cement composition. 70 to 80% of clinker would be silicates whereas the aluminates C₃A and C₄AF will form about 15 to 20% and gypsum will be around 4 to 5%.

If we look at industrially used ordinary Portland cement with grade 53 and 43, the majority of the cements will have C₃S content as dominant and C₂S content is much lower. In terms of physical characteristics, we all know that cement has a powder specific gravity or solid specific gravity of 3.15. but when we pack it as a bulk powder inside bags the density is lower which is about 1.5 to 1.6.


The weight of 1 bag of cement is 50kg and this 50 kg corresponds to 35 litres i.e. not exactly 1 cubic foot but little bit more than 1 cubic foot. So, when you use the volumetric batching bucket, commonly on job sites not having weighing facilities. The bucket exactly can hold one bag of cement. 50kg bag is designed to fill in that volumetric bucket. So, that you only need to measure the volumes of sand and stone while volumetric batching. Technically as per the concrete technology is considered, we should not recommend volume batching. We should do the weighing batching as much as possible and design of concrete should be done and kilogram per cubic metre constituent have to be actually specified in the design.

But in terms of volume batching you have that bucket exactly fitting with 50 kilograms of cement and that is the reason why we keep the cement mass at 50 kilograms in a bag. The fineness of cement is typically represented as an air permeability measurement. We call it as Blaine's fineness or Blaine's specific surface area of 300-350 square metres per kilogram that means if we take one kilogram of cement as a powder; the total surface area of the particles of constituting one kilogram will convert 300 square metres.


So the finer the cement the faster it will react obviously just like any other chemical reaction when you reduce the size or increases surface area you cause the reactivity to be higher.

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Hydration of cement




- Reaction of cement with water
- Exothermic; heat released is called 'Heat of Hydration'
- Rate of heat evolution is faster if the reaction is quicker
- Heat evolved depends on heat of hydration of individual compounds and cement fineness




What happens in cement reacts with water? We call the entire process of cement reaction with water as the hydration of cement and we all know very well that the hydration process is exothermic; heat is released, and the heat is otherwise known as heat of hydration. Obviously the quicker the cement reacts; the faster heat evolves. In general, the chemical constitution is important because it determines how much and which compound is present as each compound has its own reaction rates.

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Products of hydration of cement

- Calcium silicate hydrate (CSH) – Primary strength giving component 60% C/S = 1.5-2.0 Gel
- Calcium hydroxide (CH) – most susceptible to chemical attack 20%
- Calcium sulfo-aluminates – Responsible for early stiffening of concrete 20% Ettringite Flake



Looking at the compounds specifically C_3A or Tricalcium aluminate is responsible for setting and early strength also it has a very high heat of hydration. C_3S again reacts much faster than the other compounds except C_3A of course. C_3S also has a very high heat of hydration and it is generally responsible for the early strength gain of your cement. So, when we deal with concrete designed to achieve early strength; that early strength will be attributed to reactions of C_3S and C_3A .

The long-term strength which develops when you continue to cure concrete for long period is because of the reaction of C_2S , it is responsible for the ultimate strength or long-term strength gain and because it reacts slowly, its heat of hydration is also lower. Generally, the finer the cement faster will be the rate of heat development and quicker will be your setting and strength gain that is quite intuitive because of reduced size and more surface area.

There are more surfaces available for the reaction because of which the heat development also increases. But what are the compounds that these form upon hydration. The primary compound that forms upon hydration is calcium silicate hydrates called as CSH. CSH is only an approximate notation for this complex compound that is calcium silicate hydrate. Generally, the calcium to silica ratio in CSH is typically about 1.5 to 2.

This ratio depends on many factors that we are not going to discuss right here. The variation in compound composition of CSH can be quite extensive especially when you are talking about binders, which also incorporate supplementary cementitious materials such as fly ash or slag which when mixed with cement may actually give much different varieties of CSH that are actually getting formed in your system. But for CSH that is formed with ordinary Portland cement; typically, the calcium to silica ratios is slightly away from 1.5 closer to 2.

CSH is the phase that is almost gel like in appearance. We say it is like a gel because it is not having a very definite crystalline structure and has a sheet like structure which seems to actually stretch over a very long range. So, although the size of CSH is quite small but the agglomerations can actually be a few microns in size but if you think about CSH individually the sheets of CSH indicates a very high surface area for the material.

So if you have to do surface area application or determination techniques for CSH you will find that the actual surface area exhibited by CSH is of the order of 100000 or 200000 square metres per kilogram, and this is forming out of a material like C_3S or C_2S which has a fineness of 300 to 350 square metres per kilogram. So, you are forming a completely different compound from the hydration of the cementitious compounds C_3S and C_2S .

We will talk a little bit more about that when we get to the assessment of surface area characteristics towards the middle part of this course which will be through this BET technique. The other compound that forms in large quantities is calcium hydroxide of course it is susceptible to chemical attack, but it has a very important role to play in the concrete because that regulates the alkalinity of the concrete.

The calcium hydroxide regulates the alkalinity of the concrete and that is why it is extremely important for this compound to be present in the hydrated mixture. Then you have calcium sulfoaluminate which are forming out of the reaction of your calcium aluminates with the gypsum that is added as set regulator and these are responsible for the early stiffening of the concrete like the formation of ettringite particularly as a phase in the beginning right after the reaction of your C_3A with gypsum leads to the formation of ettringite.

This ettringite forms in the shape of needles and essentially causes the stiffening of early hydration product. But otherwise the overall volume of the constituent if you think about; it has 60% calcium silicate hydrate, 20% may be calcium hydroxide and remaining 20% is a mixture of ettringite and other forms of aluminium sulphate like AFm also called as aluminoferrite monosulphate essentially it is another form of ettringite which is sulphate deficient, it has got more alumina and less sulphur.

So you have ettringite monosulphate and you may also have other calcium aluminates hydrate products which are forming out of the reaction with aluminates directly with water. But the initial formation will be for the sulphur aluminates. If the gypsum is not added to the cement these calcium aluminate hydrates form very early during cement hydration process and lead to a condition called as flash set .


When no gypsum is present the aluminates hydrate rapidly to form calcium aluminate hydrate leading to flash setting of cement that is what we want to avoid. Since gypsum is added the first reactions end up forming calcium sulfoaluminates like ettringite which are slowly converted to sulphate deficient forms like monosulphate, but apart from that you still have the formation of calcium aluminate hydrates later on in the hydration process.


So, this is the overall composition of the hydrated cement paste CSH for the most part, calcium hydroxide to some extent and the calcium sulfoaluminate and calcium aluminate hydrates form the rest of the system.

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Relevant properties of aggregates

- Crushing strength
- Modulus of elasticity
- Hardness, impact and crushing resistance
- Specific gravity
- Gradation
- Bulking of fine aggregate
- Soundness
- Presence of impurities


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On the other hand, we have aggregates that are mostly considered to be the inert phase that we add to concrete. In most cases that is true. But in some cases you may also be sourcing aggregates from volcanic sources which leads to some reactivity in the aggregate itself. But in general when we get a sample of aggregate for use in concrete; we test several properties of these aggregate including crushing strength, modulus of elasticity, hardness, impact and crushing resistance and also specific gravity, gradation, bulking, soundness in presence of impurities All these are important characteristics test that we perform on aggregate samples

because these lend themselves to specific characteristics in the concrete. So, aggregates although they are inert, they are responsible for actually filling up the volume of the concrete and giving it dimensional stability. So aggregate gives concrete dimensional stability. What gives concrete the strength?

It is the binding of the aggregate by the paste or the bond that paste forms with the aggregate. So, essentially the strength of the concrete is contributed more by the paste system. That is why when you want to increase the strength, you have to reduce water cement ratio and make the paste stronger. The porosity of the paste depends on the water cement ratio, the lower the water cement ratio, lower is the porosity and higher is the strength of the concrete.

The aggregates are responsible primarily for keeping the volume of the concrete intact or for dimensional stability. However, if you manage to get very good gradation of the aggregate and pack the components of the aggregate well together, this will obviously contribute to the strength of the concrete because when you pack the aggregates well, you have a very little space available that you need to fill up with your cement paste and make the overall voids or porosity in the concrete to be much less than otherwise.

So, the primary agent responsible for the strength is the paste and how it binds the aggregate together. The aggregate itself is primarily responsible for dimensional stability and good packing of the aggregate enables the design of a high strength concrete mix than if you do not do a good packing of the aggregate.

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The slide is titled "Admixtures" and features the NPTEL logo in the top right corner. It is divided into two main sections: "Chemical Admixtures" and "Mineral Admixtures".

Chemical Admixtures

- Air Entrainers
- Water Reducers
- Set Controllers
- Specialty Admixtures

Basically, any chemical additive to concrete (not including paints and coatings)

Mineral Admixtures

- Fly Ash
- Blast furnace slag
- Silica fume
- Metakaolin
- Rice husk ash
- Volcanic ash
- Inert fillers such as quartz or limestone powder

A presenter is visible in the bottom right corner of the slide frame.

Apart from cement, water and aggregate we also use admixtures commonly in today's concrete and it can be classified into two types i.e. chemical admixtures and mineral admixtures. Chemical admixtures are added typically to enhance some characteristics of the concrete which otherwise are not possible by simply adding cement and water. Some of these are air entraining chemicals, set controllers, water reducers and sometimes we have speciality admixtures that are used for very specific situations.

For example, there are some chemicals that can be added to concrete, to prevent freezing when it is very cold. So, that is important because if the water turns to ice there will be no reaction between water and cement leading to hydration. Antifreeze Chemicals are one of the examples for speciality admixtures. Any chemical added to the concrete during the process of mixing concrete is called a chemical admixture.

The most common class of chemical admixtures that we deal with almost on a day-to-day basis are water reducing chemicals and as the name implies the water reducer means something that reduces the amount of water to achieve the same degree of workability in the concrete. These are the most commonly used chemicals in concrete. If you need resistance to freezing and thawing you probably would want to put air into the concrete that can be put in with the help of air entraining chemicals.

Of course, if you want to extend the workability life of your concrete, you want to use something like a retarder. If you want to shorten the period of strength gain, you will want to use an accelerator, these are set controlling chemicals that are also used in specific situations. You would not be using them as often as you use the water reducing chemical. Water reducers are the most significant to the point of view of chemical admixtures there are probably used in every concrete in today's construction world.

On the other side we have mineral additives, and these could be reactive or unreactive. Reactive mineral additives are basically by-products or waste products from other industries for example fly ash is obtained from the burning of coal in thermal power plants. This fly ash has siliceous and calcareous impurities that may lead to additional reactions apart from cement itself. The most important reaction that takes place in this system is the pozzolanic reaction that basically involves the reactive silica from pozzolan like fly ash, silica fume, metakaolin, rice husk ash or volcanic ash which combines with calcium hydroxide that is produced during cement hydration

to form additional calcium silicate hydrate. Now of course the calcium silicate hydrate we talked about is the primary binding component of your cement paste which is responsible primary for the strength of the paste.

Now the CSH additionally getting from pozzolanic reaction will contribute more to the binding although the form of the CSH may be quite different and this will be something that will be explained to you, when we actually look at the specific techniques of characterization. We can actually start picking out CSH that is of different types and of different compositions when we actually substitute cement with mineral additives.

Slag is also listed as an admixture, but actually it is a hydraulic cement that means it will react with water on its own. Slag will react with water on its own and lead to cementing reactions that form compounds similar to CSH and Calcium aluminate hydrates. Slag is not really pozzolan, but it is actually hydraulic cement bunched under the same category of mineral admixtures.

Commonly you find that people use fillers like quartz and limestone powder to enhance the particle gradation of your aggregates in concrete and also to get more fines inside to impart certain special characteristics, so inert fillers can also be used but now we know that there is sufficient research to also show that limestone is not necessarily inert some part of it, actually does react with aluminates to lead to some interesting chemistry in your system.

I will give some examples later when we talk about hydration processes that are detected through scanning electron microscope from where we will see how the microstructure actually changed significantly when you use a combination of these mineral additives and fillers.