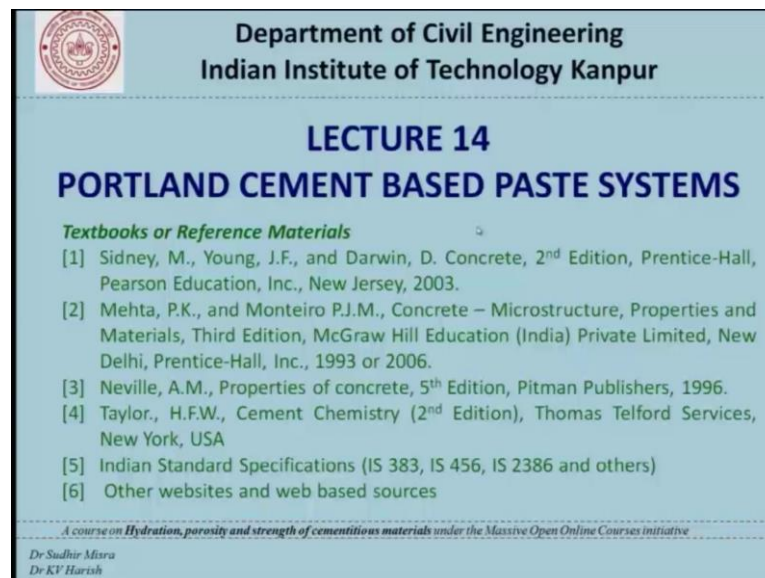


Hydration, Porosity and Strength of Cementitious Materials
Prof. Sudhir Mishra and Prof. K. V. Harish
Department of Civil Engineering
Indian Institute of Technology, Kanpur

Lecture - 14
Portland Cement Based Paste Systems

Hi. Good morning to one and all. I am K. V. Harish, Assistance Professor Department of Civil Engineering IIT Kanpur. You are watching MOOCS lecture course on Hydration, Porosity and Strength of Cementitious materials.

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LECTURE 14
PORTLAND CEMENT BASED PASTE SYSTEMS

Textbooks or Reference Materials


- [1] Sidney, M., Young, J.F., and Darwin, D. Concrete, 2nd Edition, Prentice-Hall, Pearson Education, Inc., New Jersey, 2003.
- [2] Mehta, P.K., and Monteiro P.J.M., Concrete – Microstructure, Properties and Materials, Third Edition, McGraw Hill Education (India) Private Limited, New Delhi, Prentice-Hall, Inc., 1993 or 2006.
- [3] Neville, A.M., Properties of concrete, 5th Edition, Pitman Publishers, 1996.
- [4] Taylor, H.F.W., Cement Chemistry (2nd Edition), Thomas Telford Services, New York, USA
- [5] Indian Standard Specifications (IS 383, IS 456, IS 2386 and others)
- [6] Other websites and web based sources

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Dr KV Harish

Today we will see lecture 14 Portland Cement Based Paste Systems, some of the other topics. The text books and reference materials are shown here.

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PORTLAND CEMENT BASED PASTE SYSTEMS

OVERVIEW

This lecture will provide details about the stages in the cement hydration process. In addition, the importance of setting and hardening process and the characteristics of hydrated compounds are discussed.

TOPICS

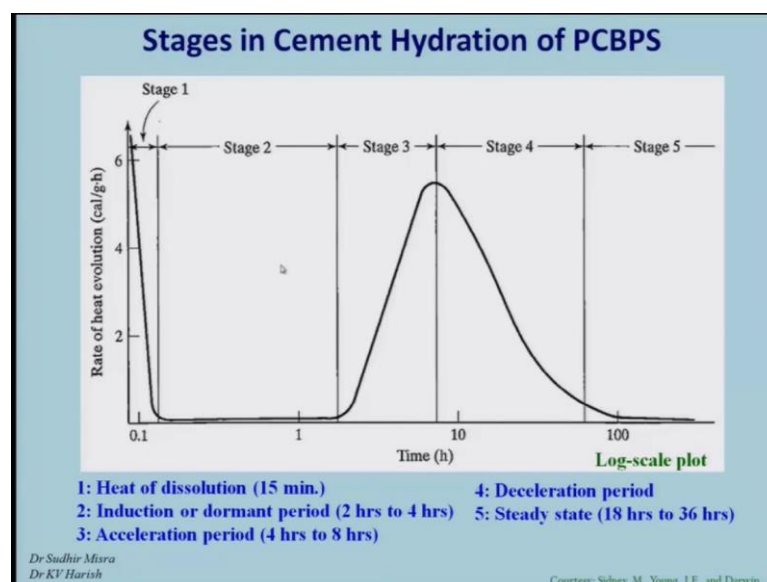
- Stages in cement hydration
- Understanding setting and hardening process
- Characteristics of hydrated compounds (MAJOR)

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So, the topics cover under this lecture are stages in cement hydration process, understanding setting and hardening process, characteristics of hydration compounds, overview of the course is as follows. This lecture will provide details about the different stages in the cement hydration process, the importance of setting and hardening process and the characteristics of hydration compounds are also discussed.


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Now, stages in cement hydration process. In the last lecture we have seen the rate of heat evolution curve, where we saw that with respect to time initially there is a decrease and

constant rate and then later on the rate of heat evolution increases further on it decreases and then it reaches a stable state. Now this rate of heat evolution curve is actually divided into 5 different stages stage one is called as heat of dissolution this usually happen during the first 15 minute from the time water is added to cement and the second stage is called as the induction period or a dormant period. Approximately it is 2 hours to 4 hours and the third stage is acceleration period which is between 4 hours to 8 hours approximately followed by the deceleration stage 4 and the final one is a steady state. So, we will see each stage in detail.

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Stage 1: Heat of Dissolution

Reaction Stage	Kinetics of Reaction	Chemical Processes	Relevance to Concrete Properties
1 Initial hydrolysis	Chemical control; rapid	Initial hydrolysis; dissolution of ions	

- Dissolution of Ca, aluminates, alkali and sulfate ions in mix water (**through solution mode**)
- Very rapid rate of reactions

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Courtesy: Saha, M., Young, J.F., and Davis

So, the first one is heat of dissolution and what basically happens is you have the initial hydrolysis, and there is dissolution of calcium aluminates alkali and sulfate ions all of which we already seen in previous lecture, the moment water is added to the cement. So, and the mixture is usually wet and the hydration process is many times defined by a through solution mode. Now kinetics of reaction is explained as chemical control and it is very rapid. And the chemical process is basically dissolution of ions or initial hydrolysis. And a basically during this period the compounds are not really formed and hence there is very minimal or no importance to concrete properties the reactions are very rapid.

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Stage 2: Induction or Dormant Period

Reaction Stage	Kinetics of Reaction	Chemical Processes	Relevance to Concrete Properties
2 Induction period	Nucleation control; slow	Continued dissolution of ions	Determines initial set

- Ca⁺⁺ and OH⁻ are being released
- Takes time to reach a critical level of ions, before Ca(OH)₂ nucleates

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Courtesy: Sahu, M., Young, J.F., and Darwin

In the second stage which is called as induction or dormant period the kinetics of reaction is termed as nucleation control, and the reactions here are very slow. And it takes time for the phase system to release ions and for the actual reaction to happen it takes some time.

So, during this time what happens is the calcium hydroxide basically nucleate and it requires certain critical level of ions, and this primarily in terms of concrete properties determines the initial set.

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Stage 3: Acceleration Period (contd..)

Reaction Stage	Kinetics of Reaction	Chemical Processes	Relevance to Concrete Properties
3 Acceleration	Chemical control; rapid	Initial formation of hydration products	Determines final set and rate of initial hardening

- Start of acceleration → **Initial set of cement**
- Initial set of cement → **2-4 hrs** from water addition
- Initial set → **No longer can mould concrete**

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The third one is the acceleration stage. In this stage the chemical reaction basically starts and the reactions are also rapid. And the chemical processes is explained as initial formation of hydration products, multiple things happen during this time the first one is a precipitation of calcium hydroxide out, and solution the second one is the calcium silicate hydroid gel starts forming on the surface of grains, third one is more and more calcium ions from cement grains go into solution.

And this basically increases the degree of hydration; degree of hydration is defined as the amount of cement grains that actually participate in the reaction. The relevance that it makes to concrete properties is that this basically defines the initial and final sets period. Some more information about the acceleration period is also provided. The start of acceleration defines the initial set of the Portland cement based paste system and usually the initial set comes between 2 to 4 hours from the time water is added to cement. The initial set is also the time at which the system no longer can be mould in concrete.

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Stage 3: Acceleration Period (contd..)			
<i>Reaction Stage</i>	<i>Kinetics of Reaction</i>	<i>Chemical Processes</i>	<i>Relevance to Concrete Properties</i>
3 Acceleration	Chemical control; rapid	Initial formation of hydration products	Determines final set and rate of initial hardening
<ul style="list-style-type: none"> ○ End of acceleration → <u>Final set of cement</u> ○ Final set → 4-8 hrs from water addition ○ Final set → <u>Gains sufficient strength</u> to walk on concrete, without casting impressions 			
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<small>Dr Sudhir Misra Dr KV Harish</small>		<small>Courtesy: Schar, M., Young, J.F., and Darwin</small>	

Now, more information about acceleration period: the end of the acceleration period indicates the final set of cement. Final set typically ranges from 4 to 8 hours from the water is added to cement and final set importance is that the cement paste should have gained a substantial strength. So, that somebody can actually work on the surface without any casting impressions.

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Stage 4: Deceleration Period

Reaction Stage	Kinetics of Reaction	Chemical Processes	Relevance to Concrete Properties
4 Deceleration	Chemical and diffusion control; slow	Continued formation of hydration products	Determines rate of early strength gain

- As C-S-H gel coats the cement grains, water cannot reach inner portions
- Hydration is diffusion controlled

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Courtesy: Sahu, M., Young, J.F., and Darwin

Now, the fourth stage is a deceleration period this stage in terms of kinetics of reaction is explained as chemical and diffusion control. And the reaction are also generally explained as slow the chemical process is nothing, but continued formation of hydration products and what happens here is that the calcium silicate hydrate gel coats the cement grains water cannot reach the inner portion very important. And finally, the hydration is going towards a diffusion controlled mode. With regard to the system properties this period determines the rate of early strength gain.

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Stage 5: Steady State

Reaction Stage	Kinetics of Reaction	Chemical Processes	Relevance to Concrete Properties
5 Steady state	Diffusion control; slow	Slow formation of hydration products	Determines rate of later strength gain

- After 48 hours, the hydration reaches approximately steady state condition
- Hydration is diffusion controlled

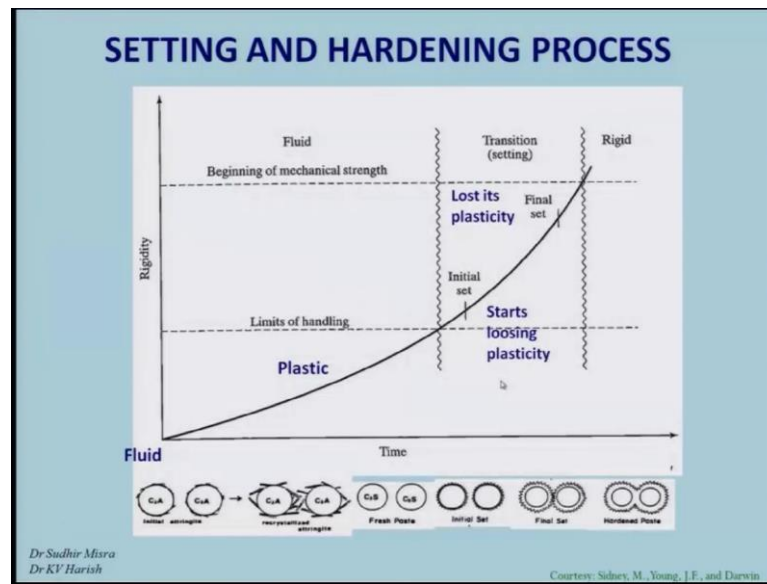
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Courtesy: Sahu, M., Young, J.F., and Darwin

Now, in the fifth stage which is called as the steady state, it is fully diffusion control continuation of the previous stage the reactions are explain to be very slow, and the chemical process it is slow formation of hydration products, and this typically happens after 48 hours the importance that this stage makes to concrete properties is that this basically determine the later age strength gain. Now little different way of understanding setting and hardening process of PCBPS systems are shown.

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So, in this figure what you find is the time is basically taken in the x axis, and the rigidity of the system is taken in the y axis. And the curve basically shows that it is ranging from 0 indicating that it is a fluid which has zero rigidity and slowly it reaches some defined strength.

So, as soon as water is added to the system, the system becomes fluid. And slowly from fluid it goes to a plastic stage. And from plastic stage it is goes to the initial set which defines the limit at which the system starts loosing the plasticity, and then it reaches the final sets where the plasticity of the system is more or less completely lost. In other words the system has gain substantial rigidity, that it can start taking loads. Now the first stage which is the fluid or plastic this largely depends upon the amount of water that is present in the cement. In such cases the system initially behaves as fluid and then goes to the plastic stage where as water to cement ratio is low or water content is low then

directly after adding the water to the mixture the mixture becomes plastic. So, there is no fluid, but it becomes plastic and from then on it goes to the other stages.

Now, in the figure you also have different levels that are shown. One is the limit is of handling the other one is the beginning of mechanical strength. So, the limit of handling is the time until which one can work with concrete many times it is also considered as the time at which the mixture is workable. And once it goes to the next level mechanical strength it is completely not workable. And the region between the initial set and the final set is called as the transition stage or setting stage. At the bottom you also see what compounds are primarily formed from the time water is added to cement to the time it gain substantial rigidity. So, at the initial stages you see that it ettringite is formed and slowly it crystallises, and then you have slowly the formation of C3S and initial set happens and then you have final set happens. And the finally, you have the harden paste where the hydration is completely defuse and control.

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Influence of setting and hardening process due to changes in the molar ratio of sulphates to aluminates

$C\bar{S}H_2/C_3A$ Molar Ratio	Hydration Products Formed
3.0	Ettringite
3.0-1.0	Ettringite + monosulfoaluminate
1.0	Monosulfoaluminate
<1.0	Monosulfoaluminate solid solution
0	Hydrogarnet

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Courtesy: Sydney Masonry Union and Darwin.

There were also other important factors which affect setting and hardening process. And one primarily factor is the amount of sulfates that is present in the system. Remember that gypsum is a main source of sulfates and Portland cements. And we have already seen in the previous lecture that the amount of gypsum is primarily dependent on the amount of aluminates, and if this proportion is not properly controlled then we will have different hydration products.

So, what is shown in this table is that in the first column, you have gypsum to tri calcium aluminate to molar ratio. In the second column you have the different hydration products that could form. So, if you see carefully for a molar ratio of say approximately 3 which is generally considered to be optimal, you get ettringite as the hydration product. Now if this ratio decreases which means sulfates are present in lower amount or gypsum is present in lower amount in that case you typically land up getting some monosulfoaluminates in addition to ettringite. And this happens typically at molar ratio of 3 to 1. Now if the molar ratio is substantially lower in other words if the sulfates are very less present in the system, in such a case you typically get land up getting only monosulfoalumainates. This happens at a molar ratio of 1. Now if the sulfate are present still lower than this value which is explained here as lower than 1, then you typically land up getting monosulfoaluminate solid solution, and typically when sulfates are absent then this molar ratio is completely 0, finally you get the product hydrogarnet.

Now we are also going to see how the presence of these sulfates or the moral ratio is going to affect the setting and hardening process.

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		Hydration age			
Reactivity of C ₃ A in clinker	Availability of sulfate in solution	<10 min	10-45 min	1-2 h	2-4 h
Low	Low	workable 	workable 	less workable 	normal set
High	High	workable 	less workable 	normal set 	Ettringite in pores
High	Low	workable 	quick set 		
High	None or very low	flash set 	C_4AH_{13} and $C_6A_3H_{18}$ in pores 		
Low	High	false set 		Crystallization of gypsum needles in pores 	

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Figure 6-8 Influence of the aluminate/sulfate ratio in the solution phase on setting characteristics of portland cement pastes. (From Locher, F.W., W. Richartz, and S. Sprung, *Zement-Kalk Gips*, No. 6, pp. 271-277, 1980.)

Courtesy: Melita and Munir

So, the figure here shows at one level at one end you have the C3A content and at the other end you have the sulfate content, and the quantity are not clearly mentioned, but generally they have referred as are low high. So, based on that you have 5 different cases, you can have a low case where in the content of C3A is generally low in the entire

Portland cement linker. And the sulfates can also be generally low, but even the both are low it can still have a molar ratio of 3.

So, in the other side what you have is what happens to the setting and hardening with hydration edge. So, here you have 4 columns, in one column you have what is happening within 10 minutes and other column you have what is happening between 10 to 45 minutes in third column you have what is happening between one to 2 hours fourth one 2 to 4 hours. So, for the case one, where you have low amounts of C3A in the entire system and you have low amount of sulfates, but the balance of C3 and sulphate are optimal. In such case the mixture will be workable at the first level.

And at the second level between 10 to 45 minutes the mixture will still be workable. And at the third level it will be less workable because ettringite start forming, and typically we get a normal set of 2 to 4 hours. And the first case is; what is a typical case that we preferred to have in the Portland cement based paste system. And in the second case we can also have increase amount of C3A, but at the same time we can also have increased amount of sulfates.

So, in such cases also the molar ratio could be optimal which means the molar ratio approximately equal to 3 where we get ettringite as a hydration product. So, in case 2 what we find is that the mixture will be workable say lower than 10 minute at the second level it will be slightly less workable, and in a third stage typically one to 2 hour you have the normal set. And remember that the normal set primarily happens because of the formation of ettringite from the view of general Portland cement based paste system. The first case and second case are preferred, primarily because there is a good balance of tri calcium aluminates and sulfates. Although in the first case it is low and the second case it is high. Now coming to the third case where C3A is substantially higher compare to the sulfates or gypsum.

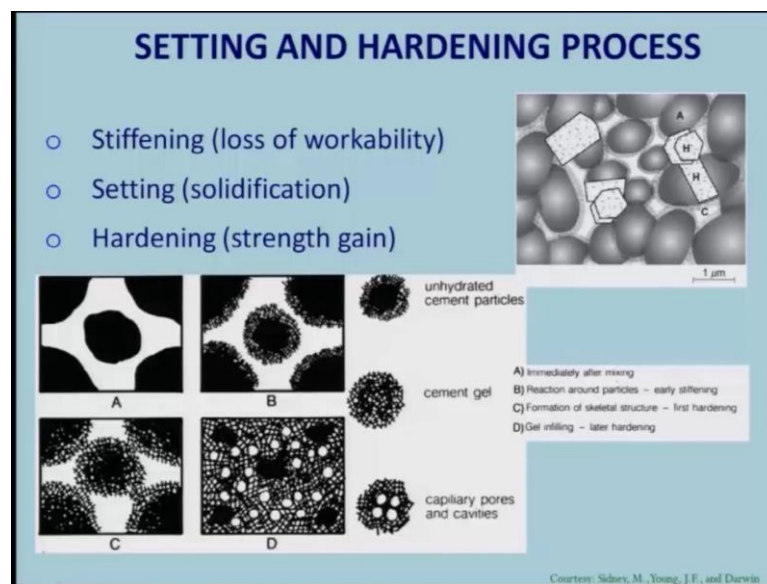
So, that is explain as high low. In such a case the mixture will be workable during the first 10 minute, whereas the second level between 10 to 45 minutes. The setting of the system happens very quickly. And remember that we have already seen when we discuss chemical reaction that we generally do not prefer quick set. So, in the case of high low case we have a quick set which happens within 45 minutes, and the fourth case where you have high amount of C3A and the low amounts of sulfate are almost 0 percentage of

sulfate. Then what you find is that within 10 minutes the entire mixture sets and this is primarily due to the formation of hydrogarnet and monosulfoaluminate and other calcium aluminates hydrates.

And remember that we do not prefer case 3, case 4. And coming on to case 5 where the C3A content is substantially low and the sulfate content is substantially higher, then what is actually required to balance in such case the mixture does not set at all it explained as falls it. So, the mixture do not set within 10 minutes it do not say within 10 to 45 minutes or it do not set at all completely.

So, the purpose of this figure is to indicate that for the regular Portland cement based paste system case one and case 2 are preferred and other cases generally are rejected or we do not preferred other cases.

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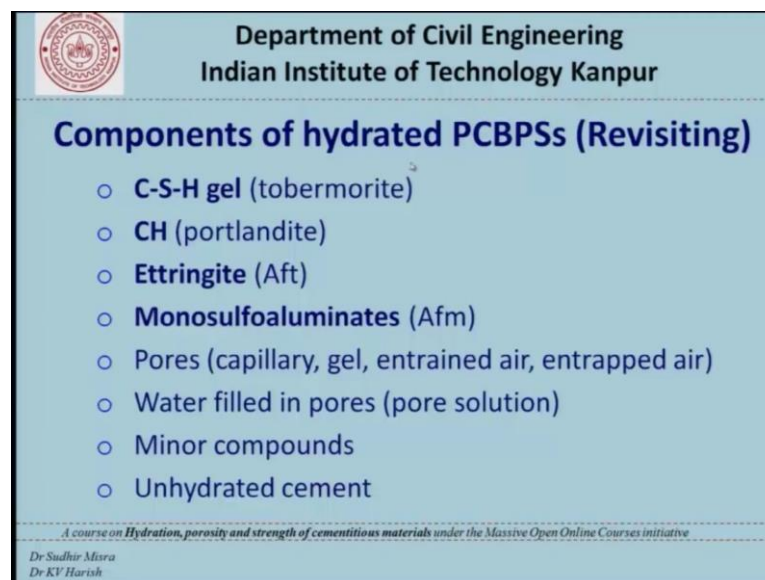



Now moving on to the next level to understand setting and hardening, the system basically under goes 3 types of changes. One is stiffening the other one is setting the other one is hardening. Stiffening is defined many times as loss of workability. Setting is many times defined as solidification and hardening is many times defined as gain of strength. And what you see in this figure is basically the water which is shown as white region and cement grains is shown as black region. And A, B, C, D, are the different stages. A is immediately after mixing, B is reaction around particles which is called as early stiffening, C is formation of skeletal structure explained as first hardening. And

finally, D is gel infilling or later hardening. You also see that these particles are explained as unhydrated cement particles which basically exist at the centre once the compounds are formed on the surface. And the hydrated compounds are basically collectively called as cement gel and with further hydration.

So, A goes to B, B goes to C and finally, it becomes D. And what you see is that you still have some pores in the system and these pores are called as capillary pores. We will see more about this information at later stage. And here also another information is parallel figure is shown for general understanding. Now the next topics are characteristic of hydrated compounds and primarily what we will see is the major hydrated compounds in the systems.

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Components of hydrated PCBPSs (Revisiting)

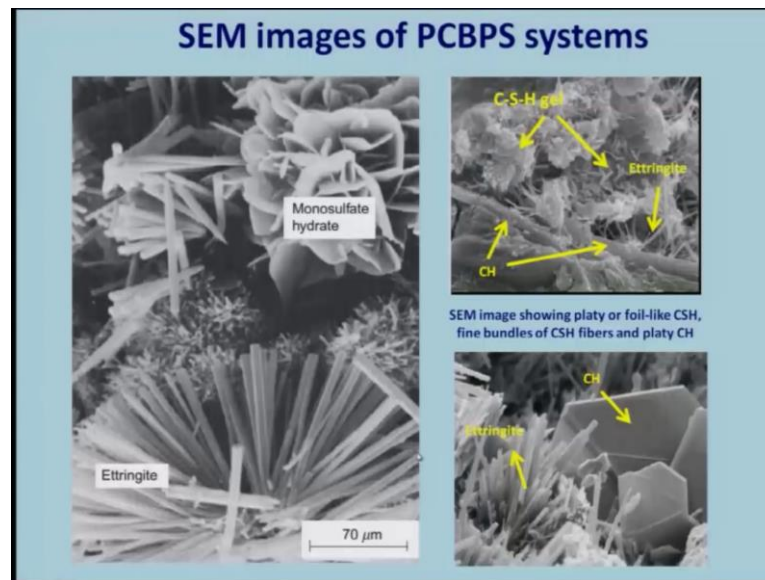
- C-S-H gel (tobermorite)
- CH (portlandite)
- Ettringite (Aft)
- Monosulfoaluminates (Afm)
- Pores (capillary, gel, entrained air, entrapped air)
- Water filled in pores (pore solution)
- Minor compounds
- Unhydrated cement

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So, we have already seen that the compounds of hydrated system include calcium silicate hydrate gel, calcium hydroxide called as portlandite ettringite Aft monosulfoalumainates Afm and you have pores water filled pores minor compounds and unhydrated cement.

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So, typically in scanning electron microscope images, what you typically find is you have different compounds like monosulfate hydrate, ettringite, and other calcium hydroxide, and these are all clubbed together. So, 3 figures are shown for easy understanding and remember that we have already seen these figures individually in the previous lectures; now coming on to characteristics of the major compounds.

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Characteristics of C-S-H gel

- Does not have a definite stoichiometry
- Structure ranges from **poorly crystalline (amorphous) fibers** to reticular network
- C-S-H gel is characterized chemically by its **C/S ratio**
- C/S ratio → 1.1-2.0; **1.5 (typical)**; depends on
 - water-to-cement (w/c) ratio
 - **degree of hydration (α)**
 - temperature of hydration
 - Impurities (primarily sulfates, alkalis)

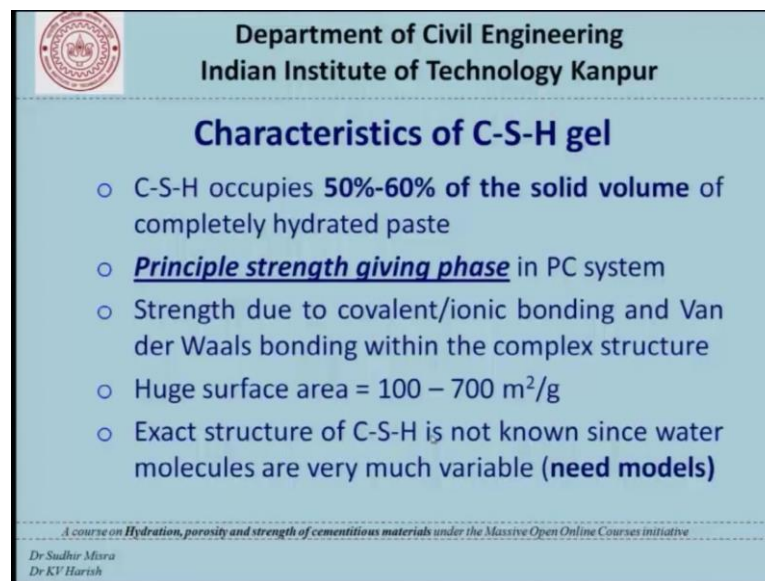
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So, we are starting with calcium silicate hydrated gel. Calcium silicate hydrate gel does not have a definite stoichiometry. The structure ranges from poorly crystalline or

amorphous fibres to reticular network. Calcium silicate hydrated gel is characteristic chemically by its C by S ratio. Remember, C stands for calcium oxide S stands for silicon oxide C by S ratio typically ranges from 1.1 to 2 and many times 1.5 is what we generally find, and the C by S ratio primarily depends on where even if parameters including order to cement ratio degree of hydration, temperature of hydration and impurity like sulfates and alkalis.

(Refer Slide Time: 22:39)



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Characteristics of C-S-H gel

- C-S-H occupies **50%-60% of the solid volume** of completely hydrated paste
- ***Principle strength giving phase*** in PC system
- Strength due to covalent/ionic bonding and Van der Waals bonding within the complex structure
- Huge surface area = 100 – 700 m²/g
- Exact structure of C-S-H is not known since water molecules are very much variable (**need models**)

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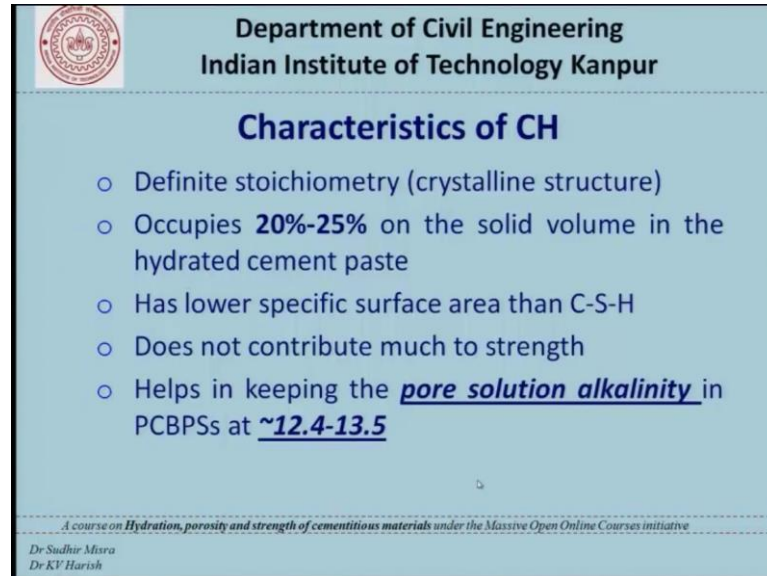
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More information about the characteristic of the gel; calcium silicate hydrate gel occupies 50 percentage to 60 percentage of the solid volume of completely hydrated cement paste. Remember that the system is never completely hydrated even after 6 months or 1 year.

But if you take a completely hydrated system we can approximately understand that 50 percentage to 60 percentages will be calcium silicate hydrate gel. And the importance of calcium silicate hydrate gel is that, it is a principle strength giving phase for the Portland cement based paste systems. And the strength is primarily due to combination of covalent or ionic bonding and van der Waals bonding within the very complex structure it has. The calcium silicate hydrate gel has a huge surface area of 100 to 700 metre square per gram and the exact structure of calcium silicate hydrate gel is not actually known primarily because the water molecules are very much variable and we actually need

specific models theoretical or empirical models to understand the structure of calcium silicate hydrate gel.

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Characteristics of CH


- Definite stoichiometry (crystalline structure)
- Occupies **20%-25%** on the solid volume in the hydrated cement paste
- Has lower specific surface area than C-S-H
- Does not contribute much to strength
- Helps in keeping the pore solution alkalinity in PCBPSs at ~12.4-13.5

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The second one is characteristics of calcium hydroxide. Calcium hydroxide has a definite stoichiometry and it is crystalline in nature. And it generally occupies 20 percent to 25 percentage of the solid volume of hydrated cement paste. Calcium hydroxide has lower specific surface area than C-S-H gel. And calcium hydroxide does not constitute much to the strength of the Portland cement based paste systems, but we have other advantages of calcium hydroxide in the sense that it helps in keeping the pore solution alkalinity approximately in the range of 12.4 to 13.5. This is extremely important from a stand point of corrosion, what is pore solution and other things we will discuss little later.

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Characteristics of Aft

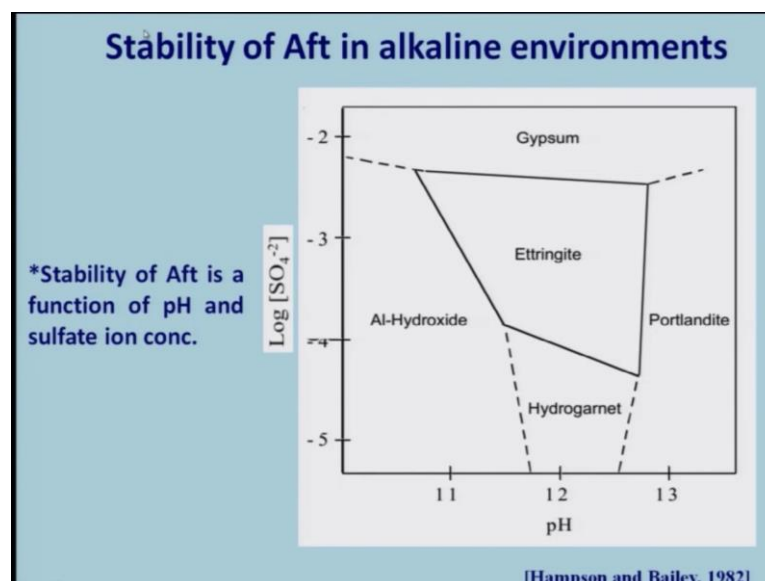
- Needles interlock and take up more water
- Contributes to the stiffening of mixture and hence, influences setting time
- Aft has some early strength
- Aft, Afm, and other minor compounds together constitute ~15%-20% of solid volume of hydrated cement paste
- Aft is extremely sensitive to alkaline environments

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Now, the third one is characteristics of Aft which is nothing, but a ettringite. They are needles interlock and take up more water. It primarily contributes to the stiffening of the mixture and hence influences setting time. Aft has some early strength; however, since the percentage of tri calcium aluminates are very low, this is not a very big factor in the strength development of Portland cement based paste system Aft, Afm and other minor compounds together constitute approximately 15 to 20 percentage of the solid volume of hydrated cement paste. And Aft is extremely sensitive to alkaline environments.

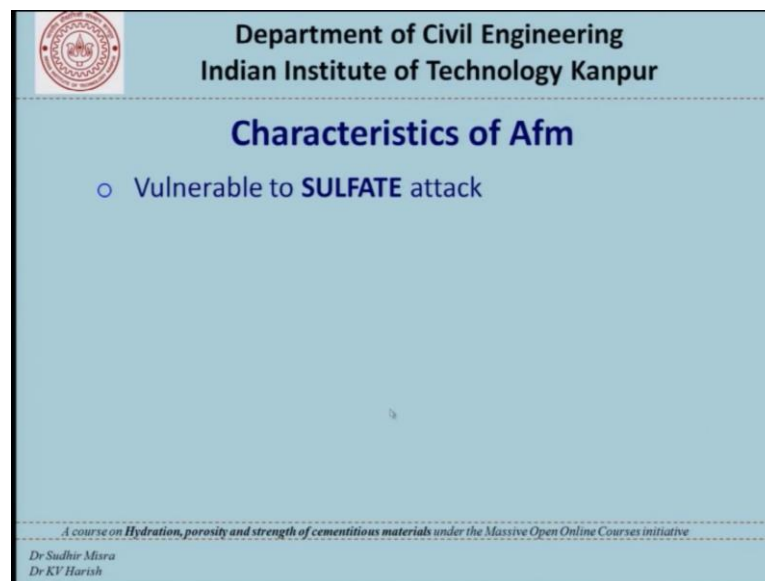
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So, figure is shown here to understand at what the sulfate ranges and what pH range ettringite is stable. So, here in the x axis you have the pH in the y axis you have sulfate concentration expressed in log scale. Remember that pH is nothing, but minus logarithm to the base 10 of hydrogen ions concentration. You see that the stability of ettringite is a function of both pH and sulfate ion concentration. So, here in this figure you have different compounds.

So, you have aluminate hydrates you have gypsum you have ettringite, you have hydrogarnet and you also have portlandite. Typically, if you see the area where a ettringite is stable, it is somewhere from here to here. So, in terms of x axis you find that a ettringite is stable somewhere between 10.8 pH to about 12.8 or 12.9 pH likewise the sulfate concentration ranges from minus 2.3 to upward minus 4.3.

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Characteristics of Afm

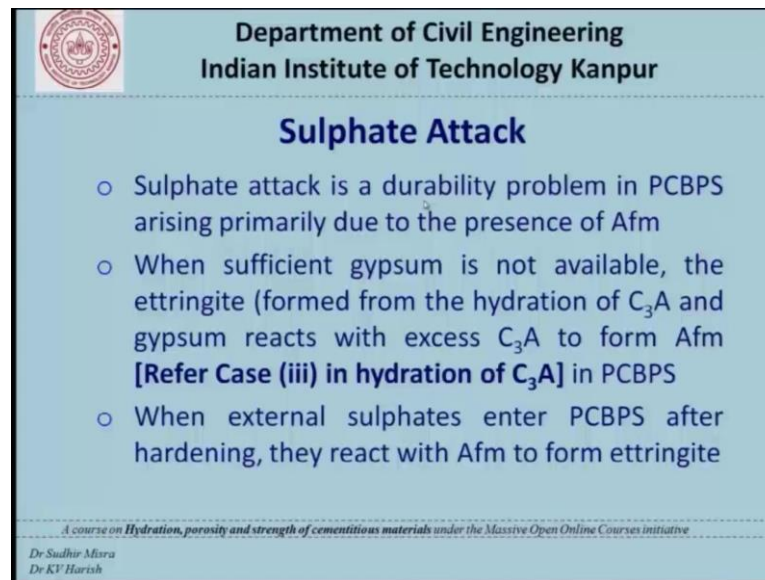
- Vulnerable to **SULFATE** attack

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Now, moving on to the fourth one which is monosulfoaluminate the characteristic of monosulfoaluminate is extremely important, and one important aspect is that we usually do not prefer monosulfoaluminate primarily because of sulfate attack, which is an important durability problem in Portland cement based paste systems. So, we will discuss the little bit about sulfate attack.

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Sulphate Attack

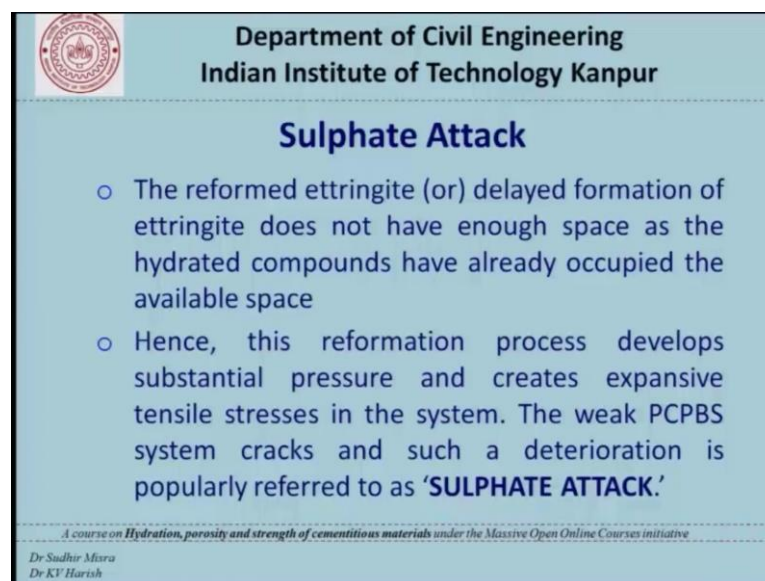
- Sulphate attack is a durability problem in PCBPS arising primarily due to the presence of Afm
- When sufficient gypsum is not available, the ettringite (formed from the hydration of C_3A and gypsum reacts with excess C_3A to form Afm [Refer Case (iii) in hydration of C_3A] in PCBPS
- When external sulphates enter PCBPS after hardening, they react with Afm to form ettringite

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Sulfate attack is a durability problem in Portland cement based paste system, arising primarily due to the presence of monosulfoalumainates. When sufficient if you get back to some of the reactions that we saw in the previous lecture, and see for which case we got monosulfoalumainates, you will find that when sufficient gypsum is not available the ettringite formed from the hydration of C_3A and gypsum reacts with excess C_3A to form Afm or monosulfoalumainates, when external sulfates enter PCBPS after hardening they react with Afm to form ettringite.

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Sulphate Attack

- The reformed ettringite (or) delayed formation of ettringite does not have enough space as the hydrated compounds have already occupied the available space
- Hence, this reformation process develops substantial pressure and creates expansive tensile stresses in the system. The weak PCPBS system cracks and such a deterioration is popularly referred to as '**SULPHATE ATTACK**.'

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The reformed ettringite or delayed formation of ettringite does not have enough space as the hydrated compounds have already occupied the available space. Hence this reformation process develops substantial pressure and creates expansive tensile stresses in the system.

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Sulphate Attack

- The chemical reaction is as follows:

$$\text{C}_4\text{A}\bar{\text{S}}\text{H}_{12} + 2\text{C}\bar{\text{S}}\text{H}_2 + 16\text{H} \rightarrow \text{C}_6\text{A}\bar{\text{S}}_3\text{H}_{32}$$

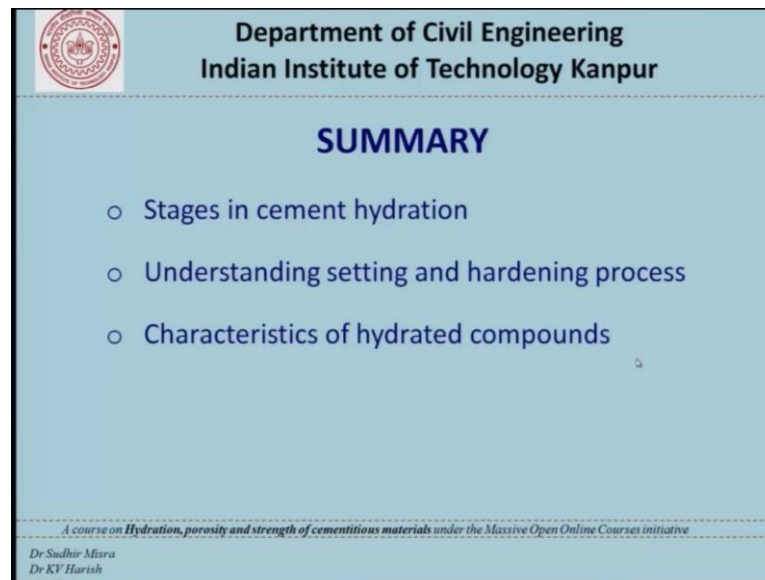
Aft *External sulfates* *Afm*




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The weak PCBPS system basically cracks and such deterioration is very popularly referred to as sulfate attack. The reaction occurring during the sulfate attack is as follows the C₄AS bar H 12 which is referred as monosulfoaluminate that is already present in the Portland cement based paste system, reacts with external sulfates in the present of water to form a ettringite. So, this reformation of a ettringite basically deteriorates the concrete because of expansion stresses created.

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SUMMARY

- Stages in cement hydration
- Understanding setting and hardening process
- Characteristics of hydrated compounds

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So, in summary we have seen stages in cement hydration process, understanding setting and hardening process and some of the characteristics of major hydrated compounds with this we are completing this lecture.

Thank you.