

Pavement Materials (Under Pavement Engineering)
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Lecture 50
Cementitious Materials (Part 2)

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WHAT ARE WE GOING TO LEARN?

- PRODUCTION OF CEMENT
- THEORY OF HYDRATION
- PHYSICAL AND CHEMICAL PROPERTIES OF CEMENT
- TYPES OF CEMENT
- POZZOLANIC MATERIALS
- GEOPOLYMERIC MATERIALS

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Hello everyone. As we were discussing in the last lecture, let us today talk about the physical properties of cement or the commonly used tests that are used to characterize the cement. So, the first test which we are going to discuss today is the initial and final setting time of cement and before we start understanding the test procedure, let us also try to understand the importance of the initial and final setting time.

So, how do we define the initial setting time? By now, we know that when the cement comes in contact with water the hydration process starts and it is a time dependent process. So, with time the cement will start to become stiff. So, if I want to use this cemented product for the construction, what do I need? I need that before I am using this product to be placed on the structure it should remain in the plastic form.

I should be able to work with this mixture of cement paste and aggregates and sand or whatever are the ingredients. I should be able to work with them properly and I should be able to mold them in certain shapes as the structure desire. So, I do not want the cement or the hydration process to be very quick initially. I want certain time availability so that I can use this mixture and I can construct this structure.

means the manufacturing should be such that the initial setting time of the cement should be at least 30 minutes.

So, the first question is if we get a cement from somewhere, how do we experiment, how do we determine or how we evaluate the initial setting time of that particular product which we have received. Now once the cement has started to lose its plasticity, it has started to harden, then what I want? Once I have placed it on that particular structure on that particular mold, now I want to compact it and then that particular structure I want to open as soon as possible.

If it is a building, I want to very quickly open the form works, if it is a pavement, I want to open it to the movement of the traffic which means that the setting of the cement after the initial setting time should not take a very-very long time which means the hardness which I want, the strength which I want should come in the concrete relatively as quick as possible after the initial setting time.

So, this period or this time which elapses between the moment I am adding water to the cement which means the beginning of the hydration process to the moment when this paste has completely lost its plasticity and has hardened so that it is now able to resist pressure, it is able to resist a load is basically termed as the final setting time.

Now these are just theoretical definition of the setting time, but we have not yet discussed that how we are going to evaluate or how we are going to find out these two setting times. The final setting time ideally should not be very high so there is a maximum limit so usually the maximum limit is about 10 hours which means that the final setting time should not be more than 10 hours because I have to open my structure to its use or to traffic for example.

Now before determining these arbitrary limits because these are always arbitrary limits So, it is not an actual value. So, before determining these arbitrary limits in the laboratory, I have to make a cement paste which has some uniform or fixed consistency because every time I change the water cement ratio, every time I change these proportions the consistency of the cement will change.

So, when I change this proportion, let us say I am taking a mix of cemented water at different water cement ratio. All these materials which I am making will have different setting times depending on the amount of water it has because again the reaction is water dependent.

So, in order to develop a standard, I want a cement paste with some specific fixed consistency so that every time I do the test, I am able to have these uniform criteria of defining the setting times. So, before I can do the initial setting time and final setting time test, I have to actually define the normal consistency

of the cement. So, this is the first step which means I have to perform an additional test to estimate or to evaluate the normal consistency of the cement.

And this is determined through the standard consistency test using a vicat apparatus. In fact, the vicat apparatus is used also for the measurement of initial and final setting time. So, the same apparatus can be first used to define the consistency then to determine the initial setting time and then to determine the final setting time.

So, let us first see that how we achieve the first criteria. What is the standard consistency here, it is that consistency when I say consistency it means amount of water by weight of the cement. So, it is that amount of water by weight of the cement which will permit the vicat plunger of 10 mm dia. So, we have a standard vicat plunger of 10 mm dia and 50 mm height, 50 mm length to penetrate a depth of 33 to 35 mm from the top of the mold.

I will show you the mold which is with me. So, this is a vicat mold, in this test what I do, I will take about 400 to 500 gram of cement, I will first mix with certain proportion of water usually that proportion is taken somewhere between 20 to 25 percent by weight of the cement, so that is the first water content.

So, let us say I take 25 percent water content or 22 percent water content by weight of the cement. So, I mix the water with the cement, I will make a paste and I will put this paste inside this vicat mold. So, I will fill this mold with the cement paste and then I will place this mold in the base of the vicat apparatus.

So, this is the vicat apparatus you can see and this is a moving arm or this is where we will actually give the penetration to the sample. So, we will put our needle here which is that particular needle, that is the plunger of 10 mm. So, this 10 mm plunger, I will attach here and then I will allow this plunger to move on the cement paste which I have just made and then I will see that what is the depth of the penetration which can be read out using the scale which is here. So, using this scale I will read out what is the depth from the top to which it has penetrated.

So, what I will do, I will make cement paste with different water content and every time I will do the test and after 2 to 3 runs, I will stop at that particular water content at which I get 33 to 35 mm of penetration from the top and that particular water content will basically be used for the or will be basically defined as the standard consistency for the source of cement I am testing.

Now in this particular test we have to remember that we have added the water to the cement to the moment we have completed this test should be between 3 to 5 minutes otherwise after coming in contact

with water the hydration process will start and this cement will start to become hard. So, we have to ensure that this test is also completed within the specified time period.

Usually, this test is done at a standard temperature of 27 degree Celsius and we have to ensure that the room in which we are doing has a relative humidity of approximately 90 percent. Because I told you that it is a 10 mm plunger, this plunger I have in my hand. So, this is the 10 mm plunger here which I will attach in the penetration needle I will attach it and then on the vicats mold which is filled with cement paste I will allow this plunger to penetrate.

So, the weight of which we use for the penetration is 300 grams as is shown in this particular picture and this is the standard dimension of the vicat mold. It is not very important for us right now the process in fact is more important.

So, after this test once we have decided for that particular source of cement what is the standard consistency or standard water content, then I will make new cement paste using that standard water content to determine the initial setting time and the final setting time. Again, we are using the same apparatus that is the vicat apparatus.

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The slide is titled "Physical Properties of Cement" and contains the following text:

- Initial and Final Setting Time
 - For initial and final setting time test, cement paste with standard consistency is prepared
 - Initial setting time in the test refers to the time elapsed from when water is added to the cement to when needle of 1 sq mm section fails to penetrate 13-15 mm from the top of the mould
 - Water added is 85% of the normal consistency
 - Needle penetration to be checked at regular intervals until the desired range is reached

Handwritten notes in red ink include:

- "85% →" next to the bullet point about water added.
- "add water + cement" with a circled "0" next to it.
- A small diagram of a needle with "1 sq mm" written next to it.

A video inset in the bottom right corner shows a man speaking. The slide footer includes a logo and the number "16".



Physical Properties of Cement

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 - Water added is 85% of the normal consistency
 - Needle penetration to be checked at regular intervals until the desired range is reached
 - For final setting time an annular needle of 5 mm dia is taken
 - Time elapsed between water addition and the moment at which needle makes an impression but the circular edge fails
- **Soundness Test (Le Chatelier Test)**
 - Presence of excess amount of lime, magnesia or sulphates are not desirable
 - Presence is evaluated indirectly by looking at the extent of expansion on standard prepared specimens

So, for the initial and final setting time cement paste with standard consistency is prepared I will tell you that what values we take to prepare the cement paste. So, initial setting time in the test it refers to the time, this is how we define the initial setting time using the vicat apparatus, it is the time elapsed from when water is added to the cement to when a needle of 1 square mm section fails to pierce 33 to 35 mm from the top of the mold. So, we will stop once that particular value is arrived at.

So, here for the preparation of sample in the first step we have determined the normal consistency but here the water which we add for the initial setting time is taken as 0.85 into P where P is that particular water content which we have determined in the consistency test.

So, what we have to do? We have to check the needle penetration at regular intervals. So, once we have the cement paste here, let us say we started our clock when we added water plus cement so we record the timing and then we have this 1 square mm needle here and I am allowing it to penetrate. So, you can imagine that initially the penetration will be higher.

Then what we will do? We will wait for some time, again because the hydration process is going on slowly this sample inside is becoming hard. So, after some time we will do the test again we will have some penetration but this penetration will be lower than the first reading and will continue to take this reading at intermediate intervals until we arrive a situation where the needle is not able to penetrate 33 to 35 mm from the top of the mold.

And remember here that this needle which we are talking about is not the same needle which we have used for the consistency test. This needle has a much smaller dia, here which you see in my hand. So, this is the needle with 1 square mm cross sectional area here and this needle we will put in the vicat penetration apparatus. We will attach it and then we will allow this needle to penetrate. And this is how we will determine the initial setting time.

Now for the final setting time again, we do not have to do any changes to the sample. We can use the same sample but now we will change the needle. We will remove the first needle and then we will attach a 5 mm dia needle. So, you can see here that this is an anural needle you can see whose dia here at the bottom part is 5 mm and a small part of the needle is piercing from the center. So, you will attach this.

Then what you will do? Again, at regular intervals you will check that when you put this needle and you allow it to penetrate, now after a certain period of time the cement paste will become sufficiently hard which means it will start to take up load. It will not allow the needle to penetrate easily and you are continuously monitoring the time since you added water to the cement so and then you did the initial setting time and then you waited for some time and in between you have used this particular needle, again you allow it to penetrate.

So, initially when the cement is not hardened enough there will be some penetration. So, you stop there you wait for some more time. Again, after some time you do the penetration test at a different location in the sample, again this might penetrate but the penetration will be smaller in comparison to what you observed in the first few runs.

So, with time same as the cement paste becomes more hard a time will come when you will penetrate this on the sample, you will observe that the impression of this edges is not there in that particular sample. Only you will see the impression of the needle which is piercing out here. So, you will not see any

impression of the circular edge and that time from the time you added the water to the cement difference between that time becomes the final setting time of the cement.

Now let us proceed to the next experiment which is the soundness test using the Le Chatelier test or apparatus. So, why are we doing this test again? This test is important to identify the presence of excessive amount of lime magnesia or sulphates in the cement. Excessive amount of this ingredients in the cement are not desirable because as I mentioned the subsequent reactions may lead to production of some crystallized products, which will increase the volume of the concrete, which will increase the susceptibility of the concrete to cracking an early failure which is not desirable.

Therefore, we have to perform some test to identify whether the cement which we are going to use in the construction is basically has desirable properties such that these unwanted materials are not present in excess amount. So, again this is an indirect test where what we do we see the extent of expansion on a standard prepared specimen.

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Physical Properties of Cement

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 - For final setting time an annular needle of 5 mm dia is taken
 - Time elapsed between water addition and the moment at which needle makes an impression but the circular edge fails
- Soundness Test (Le Chatelier Test)**
 - Presence of excess amount of lime, magnesia or sulphates are not desirable
 - Presence is evaluated indirectly by looking at the extent of expansion on standard prepared specimens
 - Prepare cement paste using 100 g cement and 0.78 P water. Immerse the assembly with glass plates and 50 g weight in water for 24 hours at 27-32 °C
 - Measure the distance between indicator points. Submerge again in water, heat and bring to boiling point in 25-30 minutes. Boil for 1 hour
 - Allow to cool and measure distance between indicators. Difference between readings should not be more than 10 mm

So, we have a mold here again, so you can see that how this looks like. So, this dimension from here to here, it is 165 mm and then you can see we are able to see a gap between these two rods here. So, what we do in this particular test we take about 100 gram cement and we mix it with 0.78 P, where P is that particular water content which we obtained during the standard consistency test.

So, 0.78 of that particular value of P we will mix it with cement and then what we will do? We will take a glass plate and we will put this mold on this glass plate. We will fill that cement paste which we have just made and then after we fill it completely to the top, we will cover it from the top using another glass plate. In this particular glass plate, what we will do, we will put a 50 grams weight. So, this is a standard weight of 50 grams which we put on this and you can tie this entire assembly using a thread.

And this assembly now what you will do, you will put it in the water. You will submerge it in the water at a temperature of around 27 to 32 degree Celsius for 24 hours. Now after 24 hours, you will remove it from the water bath and you will measure the distance between these two arms. So, these two arms you will measure the distance and you will record it.

Then what you do, you record this particular gap between the two arms let us say the reading is A and then what I will do, I will again put this assembly in inside the water bath or the container which I am using for immersing this sample. So, I will again put this sample here and then what I will do I will start boiling this particular water. I will start first heating this water, I will bring it to its boiling point in about 25 to 30 minutes and at the boiling point I will keep this particular mold emerged inside for almost 3 hours. So, this will be subjected to 3 hours of boiling. So, I will bring it to the boiling point in 25 to 30 minutes then I will boil it for 3 hours.



Physical Properties of Cement

- Compressive Strength Test

- A mortar is made by mixing 1 part of cement and 3 part of standard sand with $\frac{P}{4} + 3$ percent (by total weight of solids) water
- Standard sand comprises of 3 different grades (2-1mm; 1-500 microns; 500 microns to 90 microns)
- Take 200 g each sand grade, mix thoroughly, add cement (200 g) to this mix
- Add water of required quantity, mix and transfer to an oiled mould (each side 7.06 cm) clamped on the vibrating machine within 3-5 minutes
- Apply standard vibration of 1200 RPM for 2 minutes. Remove the mould, moist condition it with gunny bags for 24 hours (27 ± 2 °C, 90% relative humidity).



Now let us discuss about the compressive strength test. So, usually if you talk about ordinary Portland cement. So, we have OPC like OPC 33, OPC 43, OPC 53. So, these numbers which you see on the cement bag or that particular type of cement which you are using, this indicates the compressive strength of the cement material evaluated in a certain way after 28 days but the codal provisions they also mention about the minimum strength with the mortar made of that particular cement should have in 3 days, in 7 days and also in 28 days but this particular value it refers to the 28 day compressive strength.

So, how do we perform this test? We will make a mortar using one part of cement and 3 part of standard sand. And this mortar we will prepare using a water content of $\frac{P}{4} + 3$ percent where P is the standard consistency value. So, $\frac{P}{4}$ suppose if P is 24 let us say, so the value will be $\frac{24}{4} + 3$ which is 6 + 3, 9. So, 9

percent water you have to add and this 9 percent should be by weight of the total solid which means by weight of the cement plus weight of this sand.

So, this standard sand which we are mentioning here we have three types of standard sand or three different sizes of this standard sand which we define. So, we have to take sand of size ranging from 2 to 1 mm, we have to take sand of with size ranging from 1 mm to 500 microns and then 500 microns to 90 microns. So, three types of sand we have to use.


In this test what we do we will take 200 gram of each of these sand and we will mix it with cement. So, how much cement? 1 is to 3 should be the ratio so 200 total is 600 grams. So, cement should be 200 grams. So, 200 gram cement plus 600 gram sand taken equally from all the 3 sizes will be first added and mixed together and then what we will do, we will add water of the required quantity and what is that required quantity P by 4 plus 3 percent. We will mix it and we will transfer this mix to an oiled mold of standard size.

So, we have a cubicle mold which you can see in my hand, so this is the standard cubicle mold and the size is 7.06 centimeter all the sides and then we will fill it basically with the mortar which we have prepared and this mold will be basically placed on a vibrating machine. So, again here we have to remember that the mixing of water and the preparation of this, the mixing of water and then putting the sample inside the mold should be completed within 3 to 5 minutes.

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Physical Properties of Cement

- **Compressive Strength Test**
 - A mortar is made by mixing 1 part of cement and 3 part of standard sand with $P/4 + 3$ percent (by total weight of solids) water
 - Standard sand comprises of 3 different grades (2-1mm; 1-500 microns; 500 microns to 90 microns)
 - Take 200 g each sand grade, mix thoroughly, add cement (200 g) to this mix
 - Add water of required quantity, mix and transfer to an oiled mould (each side 7.06 cm) clamped on the vibrating machine within 3-5 minutes
 - Apply standard vibration of 1200 RPM for 2 minutes. Remove the mould, moist condition it with gunny bags for 24 hours (27 ± 2 °C, 90% relative humidity).
 - Demould and place it in water for conditioning
 - Calculate compressive strength (divide failure load with cross sectional area)



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So, I have a picture here to show. So, if you see that this is the vibrating machine and you will clamp your mold here, you will clamp your mold here and you will start the vibrating machine. So, vibration is usually given at about 1200 RPM, 1200 plus minus ah 400 RPMs for 2 minutes.

So, for 2 minutes you will give the vibration after completing the vibration you will remove this mold and the mole will appear to be like this which you see in the picture and then what you will do, you will keep the sample inside the mold for 24 hours and subject it to moist curing. So, the moist curing can be done at 90 percent relative humidity if you are not able to maintain 90 percent relative humidity, you can use wet gunny bags basically.

You can take gunny bag, put it in the water take it out and put it over the sample. So, you can cover this sample with wet gunny bag for 24 hours and the temperature should be 27 plus minus 2 degree Celsius. Then what you will do the next day after 24 hours is completed, you remove the gunny bag and then you unscrew this mold and you take out the mold.

So, after you take off the mold it will look something like this which you see in my hand, a small mold you will see and this mold then you will keep inside water for conditioning. So, as I mentioned the conditioning period should be 3 days, 7 days and 28 days which means you have to prepare multiple samples with the same proportion. So, a part of the sample you will keep for 3 day conditioning, a part 7 day, a part 28 days.

Then after the period of conditioning is over you will take out the sample. You will put it in a compressive testing machine as you see here you can see the sample placed here and you will just do the test until the material fails and you will record the load at failure. You know the cross-sectional area of the top of the mold. You know the load at which it has failed so you can calculate the compressive strength as load divided by cross sectional area.

And this is how you get the compressive strength test and you have to ensure that for that particular grade of cement for example if it OPC 53, then after 28 days the strength is 53 MPa and so on.

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Physical Properties of Cement

- **Fineness Test of Cement**
 - Related to the rate of hydration
 - Larger surface area of cement lead to faster development of strength and is therefore a critical parameter
 - Can be measured in two ways
 - Sieving through 90 microns. Weight retained shouldnot be more than 10%
 - Determination of specific surface area (surface area per unit weight) using Blaine air permeability test
 - Permeability cell (12.5 mm dia and 50 mm height) and plunger are used
 - Conical base connects the cell to U tube manometer
 - Perforated metal disc with 1 mm perforation used for passage of air
 - No. 40 Whatman filter paper used in conjunction with perforated plate
 - Calculated quantity of cement (say 2.8 gm) is put in the cell and compacted using plunger until it comes in contact with the cell
 - Connect the cell to the manometer using rubber coupling
 - Open stopper lid, press pressure bulb until the liquid reaches first mark AA (say)
 - Note the timing from BB to CC (2nd and 3rd marks)
 - $S = K\sqrt{t}$
 - K is the calibrated constant and t is in seconds
 - K is determined using a cement of known specific surface area

Now let us talk about the last test which we are interested to learn on cement that is the fineness test. Again, why is fineness is important because try to understand it in this way that the surface area of the cement will control the amount of water which is required to wet the surface depending on the amount of water which is required the hydration process will get affected and the rate of hydration will get affected.

So, smaller is the size of the cement particle you can try to imagine that faster will be the rate of the hydration because you have now more surface area which is available for hydration per unit volume. So, when I say surface area it is basically specific surface area which means per unit volume or per unit mass what is the total surface area of that particular particle. So, larger surface area of cement it leads to faster development of strength and is therefore a critical parameter. Therefore, we are interested to understand about this.

Usually there are two ways of measuring the fineness of cement. One is a simple method which is not very commonly used that is sieving through 90 microns, so we have a 90 microns sieve, we will take about 200 gram of cement, we will sieve it through 90 micron sieve and we will see how much material is retained. So, the weight retained it should not be more than 10 percent as per the available specification. So, this is more of an empirical way of understanding the fineness of the cement.

A more rational way of measuring the specific surface area which means surface area per unit weight is using the Blaine's air permeability test. So, unfortunately, I do not have the Blaine's apparatus here with me, so I will try to explain the process verbally to you. So, this is how a Blaine apparatus looks like. So, you see you have a U tube manometer here, this is basically the permeability cell. So, you see you have a permeability cell something like this.

So, the cell is still here only this part basically is given, this conical base, so this conical base, this is the cell this conical base basically is used to connect it to the U Tube manometer. So, this cell will be kept here. So, we have the permeability cell, this cell has again a standard dimension of 12.5 mm dia and 50 mm height. Then in this cell what we do, we will first use a perforated metal disc which is a very small disc like this and this perforation is 1 mm and this is used for the passage of air. So, we will put it here so it will rest here.

Then what we do we will use a filter paper, we will typically use a number 40 Whatman filter paper, this is a standard filter paper again of the same dimension and then we will put it over this. It will rest here and then we will add a calculated quantity of cement. I will tell you that how this quantity is calculated.

So, let us say that we have calculated how much cement is required to be used so that amount of cement is basically filled in this particular cell and it is compacted using the plunger. So, this is the plunger which you see, so we will put it inside and then we will use a plunger to compact it. So, we have to compact till the plunger comes in contact with this cell which means the top of the plunger, the plunger will look something like this. So, this part should come in contact with the top of the cell, till then we will compact it.

Now, then what we do we will keep this particular cell having the cement sample here on the manometer, using the rubber coupling. So, this black one is the rubber coupling here then we have an arrangement so this is the U tube manometer which you see. It has if a standard fluid inside it and we can maintain the pressure from here, so we have an opening here, we have a stopper lid to open and close the pressure and then we have a bulb to apply pressure here. So, this is you done using hand.

So, we will put the sample here on the top as I mentioned. We will open the stopper lid we will press the bulb until the liquid reaches the first mark. So, in the manometer you have three marks, 1, 2, 3 so first you will press until it reaches AA. And then you allow the liquid to rise and you note down the time taken by the fluid to rise between BB and CC, second and third mark.

So, you note down the timing between BB and CC, this time is used in this particular equation to calculate the specific surface area. So, specific surface area = $K \sqrt{t}$, where t is that particular time and K is the calibrated constant of the apparatus, I will tell you again how we determine the value of K.

So, K is determined using a cement of known specific surface area. So, let us say we have a standard cement whose specific surface area is known. I will using this cement I will repeat the same thing first I will place this, I will take the cell, I will put the perforated disc, I will put the filter paper, I will fill it this with this particular cement now, I will compact it as I did in the first case and then again I will put it in the manometer do the same thing see what is the time required in the standard cement to reach between BB and CC.

So, if I use the same equation $K\sqrt{t}$, I know the specific surface area here because this is a standard cement. I know the time required because I have done the test and using this I can determine the value of K. So this is how K is determined. Now the question is, how do we know that what amount of cement is required in this particular experiment.

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Handwritten notes on the slide include: $W = P \times V (V = \frac{W}{D})$, $V = \frac{W_A - W_B}{D} \rightarrow 13.53 \text{ gm/cc}$, $0.5 + 0.105$, and diagrams of a permeability cell and a U-tube manometer with labels like 'Mercury', 'Weight of the Mercury', 'AA', 'BB', 'CC', and '2.8 gm'.

So, what we have to do here we will need mercury with us, so we will have this cell here. In this cell we will put mercury to the top, then we will remove this entire mercury from the cell and we will take the weight of the mercury which is required to fill this cell to the top. So, weight of the mercury, I will note it down, let us say this is W_A .

Then what I will do, I will again take the empty cell. So, in the next step what we will do, we will take as I mentioned I will take the weight of the mercury, we will remove the mercury then we will take 2.8 gram cement then we will fill this particular 2.8 gram cement here using the same process till the lower part of the plunger touches the top of this particular cell, we will compact it, then we will place a filter paper over it, we will place a filter paper and the remaining part we will again fill with mercury.

So, this will be mercury this will be cement here. What we will do then, we will remove this mercury again after filling the top we will take the weight of this particular mercury. So, let us say that is W_B .

So, now you can see that $W_A - W_B$ will basically tell me that what amount of cement is filled here, So, if I want to calculate the volume of that particular cement this will be equal to $\frac{W_A - W_B}{D}$, D is density of the cement which is taken as 13.53 grams per CC. So, this is what is taken.

Once we have the value of V , I can calculate the weight which is required so it is equal $w = \rho \times V \times (1 - e)$ where the density is 3.15 which is standard, V we have just calculated from this experiment and e is the required porosity of the cement bed and this value of porosity depends on the type of cement for example if it is OPC, the value ranges from 0.5 ± 0.005 .

So, again for a different type of cement this value will be different and this is how we will determine the weight of the cement that is required. So, I hope that this experiment is clear to you and now you understand that how the specific surface area will be calculated and this specific surface area is again compared with the available specifications for different types of cement and we have to assure that the value meets the desired criteria.

With this we will stop here today and we have completed our discussion on some of the important properties of cement that has to be determined through laboratory investigation and previously we have also discussed in detail about the production of cement, about the hydration products, about the different chemical reactions which leads to the production of calcium silicate hydrate gel and other products which sometimes may not be very desirable. We will continue our discussion in the next class and thank you.