Admixtures and Special Concretes

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Lecture -32

Mineral Admixtures: Strength Activity test, Lime reactivity test, Mixture Proportioning and R3 test

R_3 Test method:

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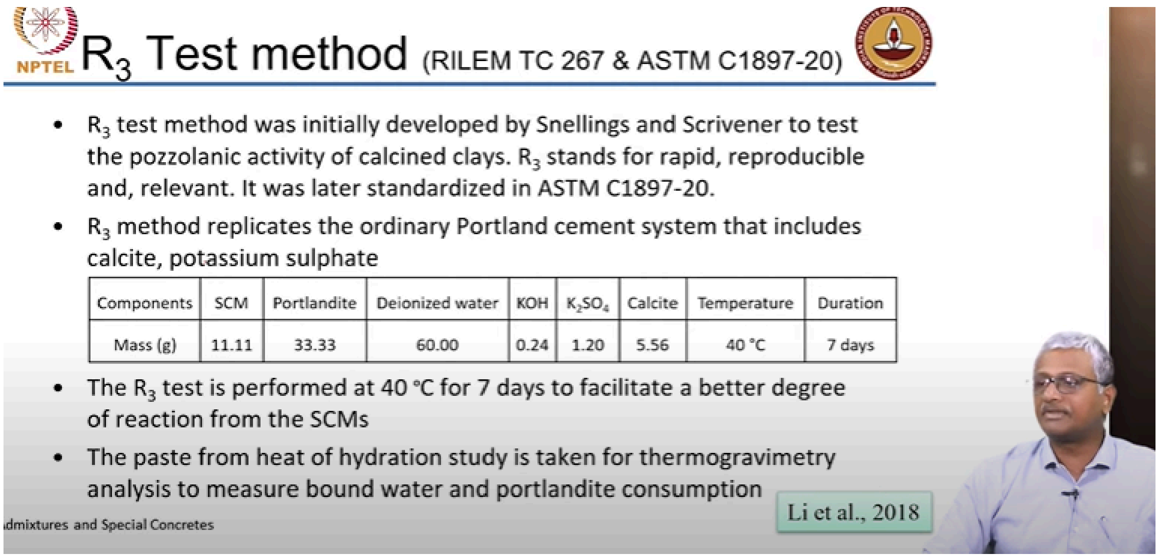

Now, based on all of these different background test methods that I talked about, there is a general consensus amongst the researchers that we need a combination of different test methods to come together to give us a clear indication of the reactivity. There is no one single test that can actually help us understand reactivity. So one needs a combination which looks at lime depletion, which looks at strength enhancement, which looks at heat evolution during the reaction and so on. We need to combine several different parameters together to really get a clear assessment of the test of the reactivity of supplementary materials. So in this case, one of the common ideas that came out of several series of tests done with RILEM TC267 and also an ASTM report was this R_3 test method. R_3 essentially means rapid, reproducible and relevant.

So R_3 was generated based on the work done in the RILEM TC committees by Ruben Slenning and Karen Scrivener and this was essentially started off as a test done for calcined clays, but later also extended to other supplementary cementing materials and this was also standardize later in ASTM C1897. It turns out that even after this R_3 test has been proposed, there have been several modifications to the same also and as I said this is a subject of much debate, but it has been universally accepted that yes we need a combination of tests to actually represent the true reactivity of the pozzolans. Now here interestingly the system does not really involve a plain lime pozzolan system. This is a modified lime pozzolan system in which what we do is, we have the supplementary material that you want to test along with portlandite or lime calcium hydroxide at a ratio of 1 is to 3.

Then you have deionized water along with a mixture of chemical ingredients like potassium hydroxide, potassium sulphate and calcite. Why are these introduced? Because we want to create an atmosphere which is similar to what the pozzolan will have in a normal cementitious matrix. When you are mixing the pozzolan along with the cement, the chemical environment will have alkalis, will have sulphates and we may have some carbonates also in the system. So in such a system what we do is, we test at a temperature of 40 degrees for a duration of 7 days. Now what we are trying to assess in this test is thermo gravimetrically how much of this lime gets consumed, how much of this portlandite is getting consumed by the pozzolanic reaction.

So the R3 test performed at 40°C for 7 days to facilitate a better degree of reaction from the SCMs. Paste from the heat of the hydration study, so we do a heat of hydration measurement using a calorimeter and we take the same paste and we do thermogravimetric analysis to measure the extent of bound water and the consumption of calcium hydroxide.

What is bound water again? Water that is bound within the structure of the hydration products that means water that you cannot evaporate from your system. By simply drying at 100°C this water does not go away, it only goes by igniting it at a higher temperature. So you determine bound water, you have to determine the heat evolution and you have to determine the portlandite or calcium hydroxide consumption. Obviously this test is a lot more evolved than anybody can do in a simple quality control lab but I will come back to that also.

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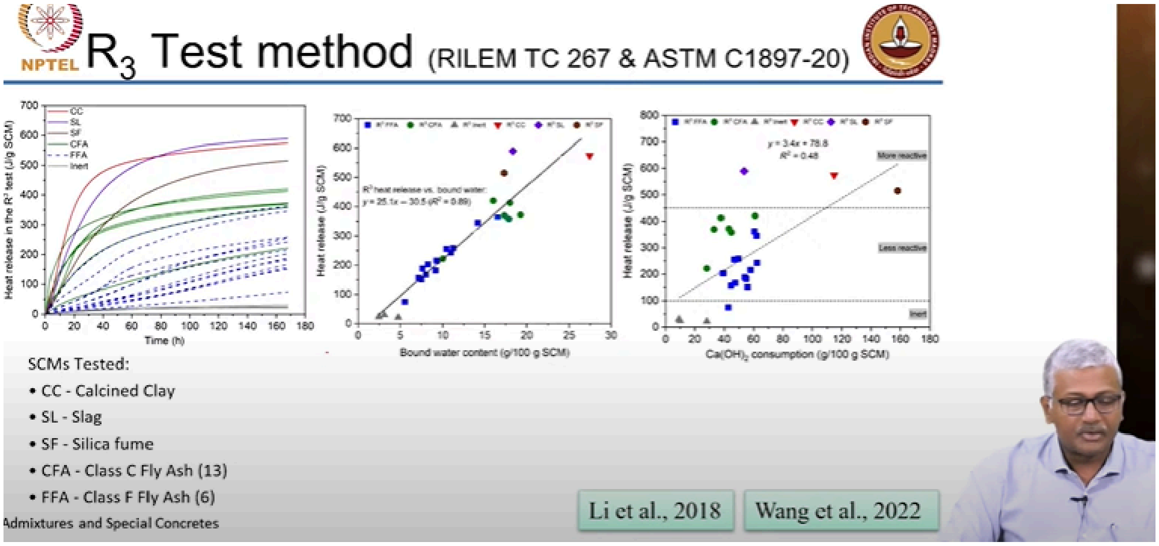

So again here what is seen is the heat release in the R_3 test plotted as a function of time. The more reactive materials are going to release heat much faster, the less reactive materials like type F fly ashes will release heat much slower as expected. So it is very clearly bringing out that distinction between inert materials which do not release any heat versus slowly reactive pozzolans like type F fly ash versus very highly reactive pozzolans like calcined clays which are right on top here. This red curve is that of calcined clay and the violet curve is that of slag, the CC and that is slag.

Now the heat release in joules per gram of the supplementary cementing material used seems to correlate very well with the bound water content. If you look at this curve here, this graph here, the linear correlation that is drawn here has a significantly high correlation coefficient of 0.89 or regression coefficient of 0.89 and what you clearly see is that there is a distinct increase in the extent of heat release with the bound water content and that is only natural. Heat is getting released because of the reaction and bound water content also goes up as more and more reaction happens and more product gets created. So there is a clear correlation between heat release and bound water content and not such a good correlation between the Portland Red consumption and the heat release. So you see that this linear trend does not produce a very good result in this case.

Now what happens is not all of the pozzolanic material gets completely dissolved in your system and reacts with the lime that is available. So Portland Red consumption there is a limit to what you are actually getting there. But what you can see is that if you can look at the extent of heat release and quantify that as 100 and below being inert materials mainly these are your quartz powder for instance.

If you take crystalline quartz as an inert material, you have less reactive material, this will probably include your fly ashes and maybe some slags but mostly just the fly ashes are type F and type C fly ash. Then you have the slags and the calcined clays which are in the more reactive system where your heat release is greater than 450 J/gm of the SCM. Here greater than 100 J/gm. So what essentially this helps you do is that you can classify your material as being inert, less reactive or more reactive but again the consumption of calcium hydroxide is not really telling you a true story here. It is not telling you a story that you would like to see with respect to a perfect correlation with your increase in the heat generation rate.

So R_3 test method generally is shown to be more reproducible as compared to the Frattini test and there is also another test method of course everybody would like to create their own test but most of these are just chemical tests which look at evolution of pozzolanic activity with more and more lime consumption. Modified Chappell test is another test which is used quite often by researchers but it shows that R_3 test has a much greater reproducibility and because of that some form of R_3 either in the original form or modified form people are employing in a large way to extend to all kinds of supplementary materials.

Mixture Proportioning:

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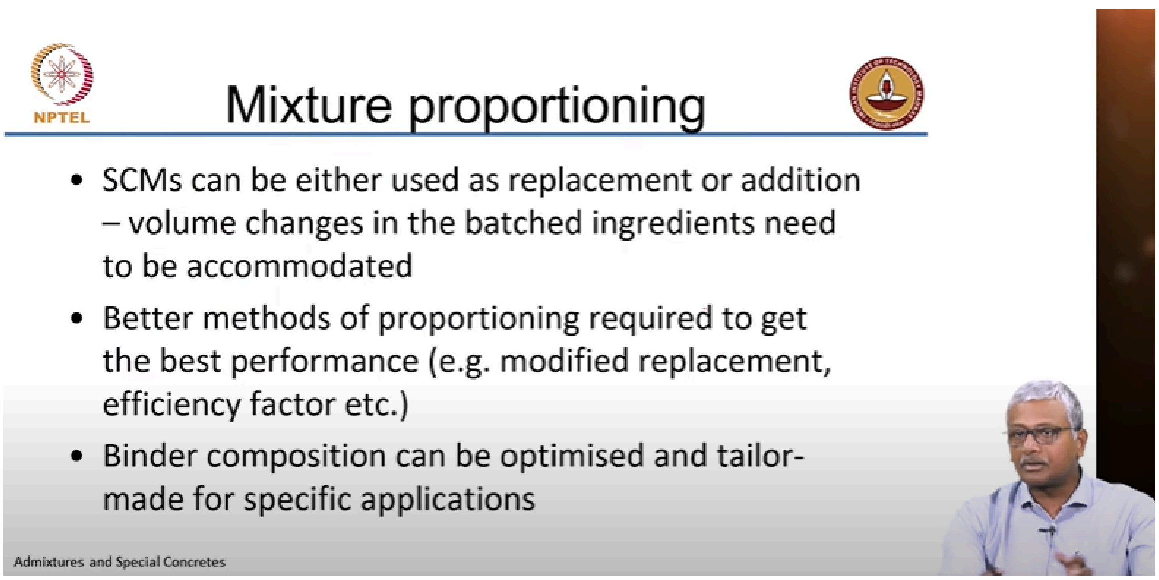

So in conclusion with respect to the activity testing of materials it is very important for us to judge the extent of reactivity shown by any mineral additive or supplementary material before we start using it in concrete. We cannot really take a material which is newly created and start putting it as a replacement of cement in concrete without assessing what it can potentially do because if the material is inert, you are not going to be able to replace cement with that material.

But what would you do with an inert material? You can use it as a filler as a portion of the fine aggregate can replace the inert material. If the material is extremely fine you can consider it as a part of the cement paste also. If the material which is a filler has size ranges of much less than 75µm then technically you do not qualify that as a replacement of aggregate you can take that as an additional cementitious material but something that is not going to react.

On the other hand the highly reactive materials like silica fume or high purity metakaolin again are restricted with respect to the extent of replacement you can do with the cement. Why cannot I replace a lot of my cement with silica fume? Heat of hydration mostly goes up because of the extreme reactivity of cement. High pozzolanic materials also will contribute to that but that may not be the reason why we cannot go for extremely high contents. There is not enough lime available in the system. Of course there are secondary effects also because many of these materials are extremely fine because of which they will increase your water demand significantly and it will not become practical to replace cement so much with silica fume or metakaolin.

On the other hand when we talk about slags which are nearly of the same particle size as cement which have a reactivity which is moderate and almost similar to cement but slightly slower you can think about replacing much larger contents also because slag is called a latent hydraulic material. Slag can react on its own with some activation. Fly ash again the reactivity is limited because of which if you are looking for concrete that produces the early age strengths your extent of replacement also is going to be limited. But on the other hand you can take advantage of the slow reactivity of the fly ash and use it in larger quantities to produce concrete that evolves less heat, reducing the heat of hydration significantly.

So depending upon the application that you want you have to make a judicious choice of what is the best mineral additive to use. Of course apart from this the availability will be a big factor which will define which additive you get to use. Something needs to be transported over a long distance. It is going to add to your cost. So it is not really something ideal that you want to use in your system.

Now when you make concrete with it, when you make concrete with the mineral admixtures replacing your cement you can consider various different ways of accommodating the volume brought in by the mineral admixtures. So you can use the material either as a replacement for the cement or keeping the cement there you add additional quantity of your material. So for instance when you are doing normal grade concrete like M20 or M30 we are looking at these materials as a replacement of cement.

We replaced cement partly. But when we are talking about high strength concrete we need a lot of cementitious material in there. We are not going to be replacing cement. We are probably maximizing cement.

How much is the maximum quantity of cement that we can use for building purposes? 450 kg/m^3. As per the IS 456 we cannot exceed cement content of 450 but in certain instances for let us say for producing self-compacted concrete you may not be able to do just with cement. You have to extend it using mineral additives. In such instances we use this as an addition and not as a replacement.

Now what will happen as a result of this is that your volume adjustment has to be done for the concrete because concrete is always designed for a given volume. You design concrete for 1 cubic meter and what is the approximate mass of that $1m^3$? What is the mass or unit weight of 1 kg/m^3? 2400 kg/m^3. Each cubic meter of concrete weighs 2400 kg.

But what will happen is if you replace cement let us say 50 kg of cement is replaced by 50 kg of fly ash. What is going to happen to the volume of the system? Which is denser cement or fly ash? Cement is denser. Cement has 3.1 density whereas fly ash has about 2.2. Siliceous materials are between 2.2 and 2.6. So you are replacing heavier material with a less dense material. So what is going to happen to the volume? It goes up. The volume of the paste is going to go up. So what will you do now? How will you compensate? You will have to probably look at other badged ingredients like fine or coarse aggregates. You are removing some volume from that to keep the overall volume the same.

But now your system has changed. Why? Because earlier you had a certain volume of paste to the volume of aggregate ratio. By replacing cement with a less dense material you are upping the volume of paste and to keep the total volume constant you are lowering the volume of aggregate. So that will produce some effects in your system. What effects will it produce? Strength may or may not get affected depending on the water cement ratio and how you design your mix. But shrinkage is going to be affected. Strength may be affected because you have more paste and less aggregate. So you have to be careful in your design methodology to not exceed or rather change this paste to aggregate ratio significantly if you are looking for similar properties. You can always design for other properties but if you are looking for similar properties as that with plain Portland cement you have to do it more judiciously.

So there are other ways of proportioning also recommended like modified replacement or efficiency factor method. Efficiency factor is not easy to apply on site. So what happens is in a modified replacement you consider part of the mineral admixture as a replacement of the system, part of it as an addition. So let us say you have 400 kg of cement and in mix 1 and the second mix you put 32 kg of cement and 150 kg of fly ash. So what I am trying to say is part of this 150 is taken as a replacement for the cement, part is taken as an addition. So instead of designing with exactly 400 cementitious materials now I am designing with 470 cementitious materials. So I am going to alter my design and make sure that I am able to get the same hardened concrete properties. Efficiency factor tells you that if I replace 1 kilogram of cement with 1 kilogram of fly ash, what is the efficiency of that 1 kilogram of fly ash? Does it produce 50% of the cement strength or does it produce 100% of the cement strength? So that essentially implies I can replace the same mass of cement by the pozzolanic material and still get the same performance. It is not easy to predict.

First of all it will depend on the age at which you are testing your performance because fly ash 1 kilogram of it as a replacement for 1 kilogram of cement may produce the same equivalent strength at 90 days but at 28 days it may not do that. So the efficiency factor is a little complicated to apply in practice. For the most part people simply do the simple replacement or simple addition that means replacement of the cement by volume or by mass. Now I have said two things here: replacement by volume or replacement by mass.

If you do replacement by volume what will happen? So I am removing let us say 400 kg of cement is my overall cement content in the mix 1. In mix 2, I have 350 cement and 50 kg equivalent volume of fly ash. What do I mean by that? I have removed 50 kg of cement and whatever volume that created in my system that much equivalent volume of fly ash I added. So what happens to your paste to aggregate ratio? It does not change because you are volumetrically replacing the system.

But again in the field if you think about it, it is a lot more complicated because you need all these values. You need to understand the specific gravity of each of the ingredients. You need to have some idea about working with volumes. That is why in the field people generally work with mass replacement. Mostly we work with mass replacements or mass additions of the cementitious system with fly ash or slag or any other mineral admixture.

So it is very important for us to keep track of how this will affect the overall characteristics because it is going to adjust your paste to aggregate ratio. So when you see the results and start comparing make sure that you are able to clearly understand what is the impact of increasing the volume of the paste on your system. Very often we see that when shrinkage results are given comparative shrinkage performance of a plain cement based system or pozzolanic system is given. Very often this issue of change in paste to aggregate ratio can lead to a major change in the way that you interpret the results or can lead to a major change in the way that the performance of concrete with respect to creep and shrinkage actually happens.