

Admixtures and Special Concretes

Prof. Manu Santhanam

Indian Institute of Technology Madras

Department of Civil Engineering

Lecture -21

Chemical Admixtures: Understanding Concrete Rheology - Part 2

Recap:

(Refer slides of previous lecture)

So, we were talking about different types of rheological models. For purposes of approximation concrete technologies often find it quite easy to adopt a simple linear model called the Bingham model. But we saw that very often we have to explain higher order issues because of the behaviour of the mix at different strain rates. In some instances you get a shear thickening behaviour and other instances you get a shear thinning behaviour. So, because of this we need to adopt the right approaches to design our mixes to get the kind of property that we desire. As we discussed in most cases when we talk about concrete we would like to get a shear thinning behaviour so that concrete at high shear rates is able to flow fairly easily with a low viscosity. But when it comes to rest it builds up the internal viscosity and that build up is also related to what we call as thixotropic property of the concrete. Now, when we get into the aspect of understanding viscosity modifying agents later we will understand that this viscosity modifying agent actually causes the concrete to exhibit thixotropy. So, the thixotropic property is essentially introduced by the viscosity modifying agent. But I will come to that after we finish talking about the different aspects of rheology.

So, as we had been discussing, the shear yield stress is related to the extent of shear stress required to initiate the flow. So, put in simple terms it is the amount of stress that you need to put to get the aggregates moving past each other. If you have aggregates which are locked by intergranular friction you need to put in some stress to get them moving past each other. So, that is essentially related to your shear yield stress and plastic viscosity is once the concrete starts flowing the resistance to that flow is the plastic viscosity and that is governed by both the viscosity of the paste and the amount of aggregates that are present in the system because the paste is essentially carrying the aggregates along with it. Okay, this is again very important when you think up of an application like pumping.

In pumping, what are we doing? We are applying pressure to push concrete through a long pipe and there are problems like frictional forces of the pipe which are restricting the

amount of concrete that can be formed. So, a concrete which has a low viscosity will tend to carry itself quite easily through the pump as opposed to concrete which is highly viscous. Okay, there are several instances when pump blockages are reported in actual practice. So, pump blockages typically happen when your concrete is losing its slump or when the concrete becomes stiffened because of a delay in the process of pumping and so on. And in some instances it could just be because your pumping heights are too much, your pumping across very long distances both horizontally or vertically and as a result of this you may be generating too much heat while pressurizing the concrete to go through the pump.

What is the world record for concrete pumping in terms of vertical height? I do not think there is a problem in guessing the answer. In Burj Khalifa they pumped up to 600m vertically and this was self-compacting concrete. So, they had to ensure that this concrete does not segregate because the pressures were quite high on the ground level. I think we have a discussion on that when we come to the SCC chapter also. But you can imagine that the concrete properties have to be designed in such a way that there is no segregation. Secondly, the frictional losses are not so high that will lead to a complete reduction in your flowable properties. So, concrete has to be self-compacting to the point it comes out at 600 meters height. So you can imagine the difficulty with that.

And we then discussed how the rheology of the concrete system changes, right? While it is getting mixed in the plant, when it is transported to the truck, carried to the job site, right? And all of this has a very significant correlation with the fundamental rheological properties.


And these rheological properties we described as being of the static nature or dynamic nature. Dynamic because you need to start moving concrete from rest, that is the static nature. Dynamic implying that concrete is already mobile and you are just pushing it out to fill up the formwork, right? So the dynamic yield stress values are generally lower than the static yield stress. The curves going up and down when you increase and decrease the shear rate are not coinciding which means that there is some hysteresis in the system and that hysteresis in terms of rheology is termed Thixotropy. I also briefly described how Thixotropic behaviour is typically assessed in the laboratory. You increase or ramp up the shear rate and every point to which you ramp up the shear rate you hold it constant for some time and then you ramp it up further and so on and so forth. And for each of these shear rates you then measure the shear stress, right? Or torque, from torque you get the shear stress. So the shear stress is highest in the beginning but then as you maintain that same shear rate it continues to drop just like what you saw in the stress growth test. So in the stress growth test we saw that as you increase the shear or rather as you increase the time of maintaining that shear your stress reaches a maximum value and then it starts to come down to a stable value, right? And that is what we are seeing here that the stress goes up maximum and then comes down to a stable value. Of course you

do not keep your shear rate maintained for a long enough time to really find out what the shear value is. But the objective here is to understand by plotting the shear stress versus shear strain rate what is the trend in the up and down curve. So you increase the shear rate and then you start decreasing and what is this trend? So the trends that exhibit the thixotropy obviously will have an area between the up curve and the down curve, okay? The larger the area the greater the thixotropy that is exhibited by the system. So if you do the same test on clays you will get a very similar result. So clays also have this aspect of internal structural build up and that we are very familiar with, right? You mix water with clay and you start applying some pressure. You can mold it in any shape you want. But after you mold it, it stays in that shape. It does not start flowing again. Unless your water content is above a certain value your system becomes stable, right? So that is basically thixotropy. It maintains its shape when you remove the pressure.


So we also looked at how this thixotropy actually affects the buildup of internal stresses when the concrete is getting transported from the plant to the job site. So all of this is very important for us to understand what could be the loss and slump that happens over the given time period. And in this connection I described earlier this approach that is adopted in North America called the verifi approach where they are actually able to assess the slump or the fresh concrete properties inside the truck as the concrete gets delivered to the job site. And that can be well correlated with the type of fresh properties that you need at the point of discharge. And you can use that system to actively control the rheological properties of your system. Now of course when we discuss self-compacting concrete or 3D printed concrete the aspect of rheological control will become all the more clearer. Right now we are only looking at how this is important from the perspective of understanding the way that viscosity modifying agents actually work.

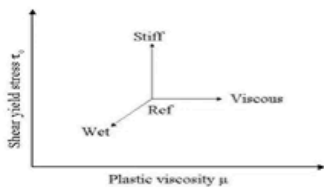
Rheology Contd:

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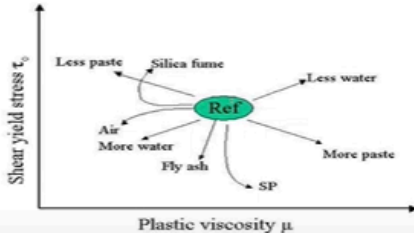


Understanding rheology






Qualitative description



Effect of mixture parameters

Wallevik (Iceland) and Domone (UK)



So let us move on. So in terms of concrete as I said concrete technologists often hide behind the linear relationship which we call the Bingham model. We are happy with that but if you talk to a real rheologist for instance in the civil engineering department if you are in our group we are happy with the Bingham model. But if you cross the corridor into the transportation group they are very strict about their understanding of rheological properties of materials because asphalt is a very interesting material from the rheology perspective. But the nonlinearity that comes with asphalt has a lot to do with the polymer type nature of asphalt because the rate of loading temperature has much greater effects in viscoelastic materials like polymers which include asphalt rather than concrete. Temperature may have some sort of an effect but it is not going to affect concrete as much as a viscoelastic material like asphalt. Of course concrete is also viscoelastic but the scale of viscoelasticity is much different as compared to asphalt.

Nevertheless looking at the two parameters that we generate with the Bingham model, what are those two parameters? Shear yield stress and plastic viscosity. So let us look at shear yield stress versus plastic viscosity qualitatively. I am not giving you any numbers here. Let us say this is your reference concrete when I say that I am making the mix more viscous. I am basically moving along this axis. I am moving along the viscosity axis without changing the yield stress much. When I say I am making the mix stiff I am moving along the shear yield stress axis. This is basically moving from a finite slump concrete to no slump concrete. What kind of applications will no slump concrete be used in? When you are concreting slopes you want to have reduced slump. What else? Roller compacted concrete where you have to apply pressure by roller compaction concrete does not need to have a high slump there so you can work very low cement contents and so on. On the other hand when I say I am making the system wet I am actually moving down both axis I mean reducing both yield stress and plastic viscosity. Add water to your system that is what it will end up doing. But if you change the paste to aggregate ratio you may not always get this kind of behaviour. Because paste content can be changed by not changing the water content also you can simply add more cementitious material. So your system will become more viscous but it will not reduce yield stress significantly. That is how you try to understand what happens when mineral or chemical admixtures are added to a reference concrete. So let us say this is your reference concrete here. If I add more water I am moving in that direction because my concrete mix gets more and more wet. When I reduce the water I am moving in the other direction I increase both yield stress and plastic viscosity. When I increase the paste I am going to increase my viscosity but at the same time what I am doing now since your paste is going up your aggregate is coming down. So intergranular friction is also coming down so you generally tend to reduce the yield stress as you increase the paste content. So less paste obviously is in the other direction: increase in shear yield stress and lowering of viscosity.

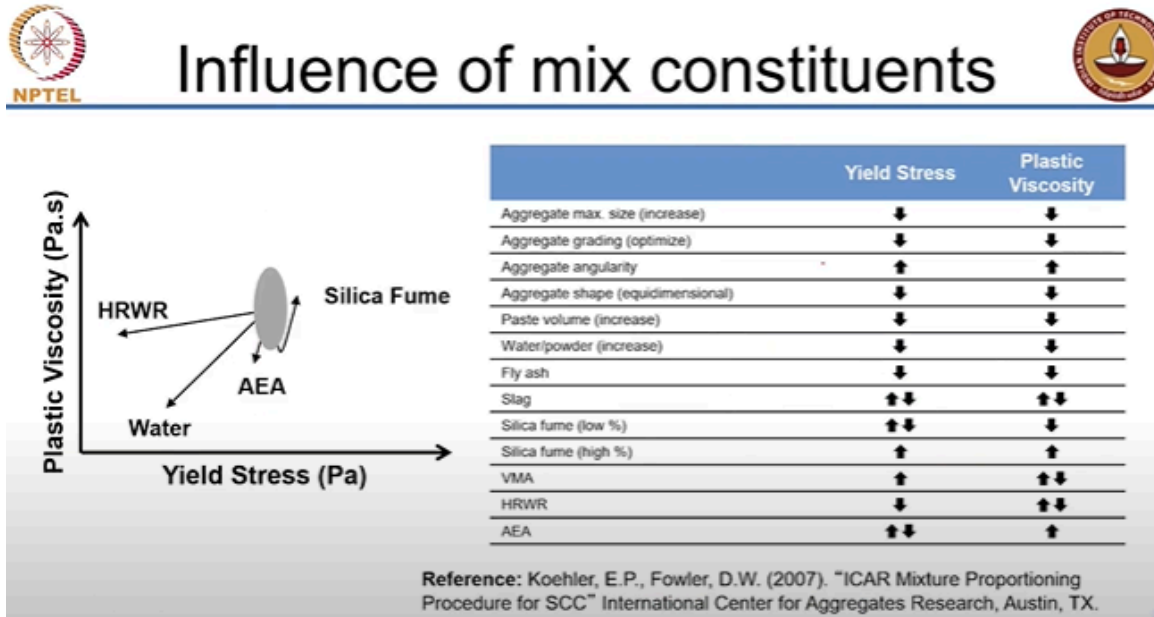
Now the other effects such as air entrainment, use of fly ash, silica fume, super plasticizer will depend a lot on the type of concrete that is being used. But for most normal concrete applications when you use a super plasticizer you will reduce the shear yield stress which is obvious because as you put the super plasticizer you are increasing the slump. However if you put too much of super plasticizer you will either get segregation that is a different property altogether or you may actually end up getting stickiness in the concrete and that stickiness may be resulting from the increase in viscosity but those are not effects that you will see all the time. When you use fly ash assuming that fly ash particles are how they should be, that is spherical particles of the same size as cement when you replace cement with fly ash you expect that the slump will go up. It may or may not affect your viscosity so that is why the slope is too small. You are basically reducing the yield stress but your reduction of viscosity may not be significant. In fact you may sometimes see an increase in the viscosity because if the fly ash is very fine then the volume of this paste, volume of the powder is going to go up.

What is the density of cement and fly ash? Cement density is how much? 3.15 right fly ash? Fly ash is around 2.2. So let us say you remove 1 kg of cement and put 1 kg of fly ash. The volume of the powder is going to go up because now you are filling up with the less dense material. So since the volume of powder goes up without changing water content that is equivalent to going for more paste content. So you may actually end up increasing your viscosity if the fly ash is very fine but for most cases you do not get that action.

Silica fumes are very fine particles right so if you replace cement with silica fumes without altering your water content or using a super plasticizer you are likely to see a major enhancement in your yield stress that means your slump is going to go down significantly when you add silica fumes. So the idea of looking at these two parameters can conveniently explain what happens to your concrete mixture as you are trying to alter the mix design right and this way you can actually have an assessment about what should be the constituents required for specific applications, that is the idea of understanding rheology.

Kohler and Fowler (Influence of mix constituents):

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So some more descriptions from a paper by Kohler and Fowler from 2007,, so here again what they have looked at is use of air entrainer high range water reducer water and silica fumes of course you have to see that the axes are interchanged. X and Y axes that I showed you previously here they plotted in different scales. So again here what they have looked at in this paper is the effect of various different things including size of the aggregate, grading, angularity, shape and so on and so forth and also the other mixed constituents like paste, water, fly ash, slag, silica fume, VMA, high range water reducer and air entering agent. So it is a mixed bag that you get here okay but again what I wanted to tell you is that this is dependent a lot on the kind of concrete you are designing normal concrete or flowable concrete or 3D printed concrete rheology needs to be understood separately for each of these systems. Basic behavior is not going to get altered much but you need to understand rheology specifically in order to be able to design concrete with these systems.

Rheological measurements:

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Geometries for rheometers



- Coaxial cylinder – only for paste
- Parallel plate – Ok for paste and sometimes mortar
- Cone and plate – not used
- Vane – commonly used for mortar and concrete



Rotational Rheometry

Admixtures and Special Concretes



So let us look at how rheological measurements are done. Now the classical definition of rheology is that you have two parallel plates right which are moving past each other at a given strain rate right and then we plot the shear stress versus the strain rate and get the fundamental rheological behavior. Now this is simple for something like water or oil okay but then when you start working with cementitious suspensions and more complex fluids it becomes a little difficult to always have the same geometry okay.

So what we generally tend to use is a coaxial cylinder geometry. So as the name implies you have an outer cylinder and you have an inner cylinder okay and the inner cylinder is rotating whereas the outer cylinder is fixed or alternatively inner cylinder may be fixed outer cylinder is rotating you can do both ways but generally it is taken in this manner. So your paste is filled up between the outer and inner cylinder. So what will happen initially is that when you try to spin your, move your spindle the resistance because of the initial shear stress of the paste will control it but after a certain point the shear will be sufficient to overcome that resistance and then you will start spinning the spindle as you keep on increasing the shear rate your shear stress will keep on increasing and you will get what is commonly called as a Bingham behavior right depending upon the type of mix that you have. So that is a coaxial cylinder geometry.

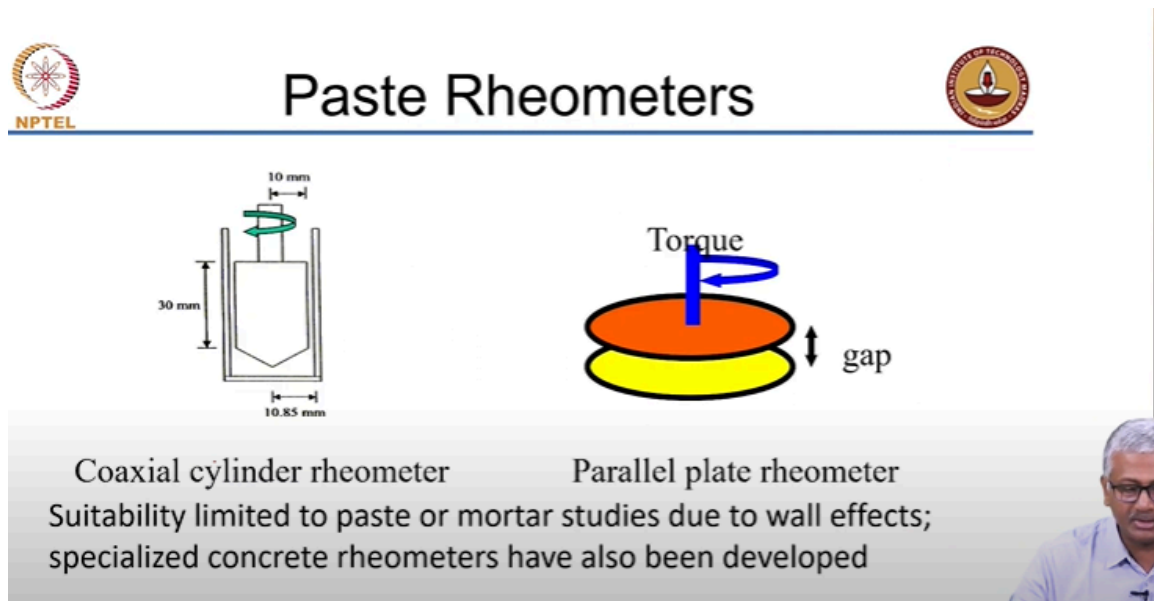
A parallel plate geometry is where you have a top plate and a bottom plate and the bottom plate is stationary while the top plate is rotated okay. So what we try to study is what is called rotational viscometry or rotational geometry. This is not exactly the same as this translational viscometry or geometry where you are actually moving the plates past each other in a linear direction but here it is a rotational geometry. So the relationships are not exactly the same as what you have with the translation. So because if you imagine if you have two plates and you are moving them past each other there is

only shear involved right but when you are having a plate like this and you have a plate on top which is rotated there will also be some sort of a normal stress on the top plate right. There will also be a normal stress in the top plate. This is not purely a shearing condition. So this is not a classical definition of viscosity but it is an indirect use of viscosity.

Okay so parallel plate is also used generally for paste and sometimes mortar. Why am I saying this coaxial cylinder and parallel plate are mainly used for paste but not really for mortar or concrete? Why? Yeah so the space between the plates or the cylinders may be so small that having an aggregate particle of greater than a vertical size may actually affect your result significantly. So you will not be studying rheology anymore. It will be dictated a lot by the intergranular friction.

Paste Rheometers:

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Paste Rheometers

Coaxial cylinder rheometer
Suitability limited to paste or mortar studies due to wall effects;
specialized concrete rheometers have also been developed

Parallel plate rheometer

Torque

gap

Now there is also a cone and plate system which is generally not used for cement paste or cementitious materials. What is most commonly used is vane geometry. I will just show you these geometries. It is commonly used for mortar and concrete because just like your soils where we use the vane shear test in concrete or mortar also you can put the vane and cause the vane to rotate. The only problem with that is that the relationships between the torque and the shear stress are not going to be as easy to establish as is the case with your coaxial cylinder or parallel plate geometries.

So just to show you this again, so coaxial cylinder geometry is shown here. As you can see the gap here is less than 1 mm. The gap is less than 1 mm. That means if your particle

sizes, generally if the particle sizes are less than the gap divided by 10 then it is okay. If the particle sizes are less than one tenth of the gap then it is fine. But if it is more than one tenth of the gap then your fluid flow between the plates or between the cylinders is not going to be the laminar kind that we are needing for defining the actual relationship between stresses. And then you have other friction and all dominating. That is going to be a problem. So if you choose cement, the maximum particle size of cement will be around $100\mu\text{m}$ but most particles will be less than $45\mu\text{m}$, so it is okay with cement. But when you start moving towards mortar, your aggregate sizes unless you control them to be less than $300\mu\text{m}$ even then you are in a situation where this ideal rule is not really satisfied.

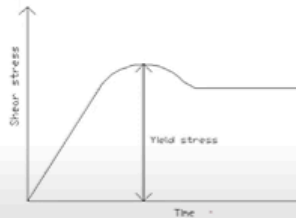
Similarly, here the parallel plate geometry gap is typically very small. Why cannot we choose just a large, why cannot we increase the gap? Interlayer friction? So the objective is to get the paste to shear. So when you are increasing the gap what may happen is since the bottom plate is rigid and the top plate is rotating, you may only get a small region in the top to start shearing. You may not actually translate that shear into the entire volume. So you will not be actually assessing the properties of the full system. So that would be a problem. That is why you cannot really have a very large gap because your flow relationships cannot be established properly there.

When you start introducing larger particles in mortar or concrete, you get what are called wall effects. Wall effects simply means that if you have a fluid in a cylinder, if you have suspended particles in the fluid like aggregate for instance, the aggregate effects close to the walls of the cylinder are going to be quite different as compared to the aggregate effects away from the wall of the cylinder. So there will be some forces caused because of the particle sizes near the wall of the cylinder which will start dominating and produce other effects which are not easy to account for and that is why doing a rheological measurement on mortar or concrete becomes quite difficult. Not impossible, there are mortar and concrete rheometers that are specifically designed for this purpose but those do not have the same easy conversion to a fundamental relationship as you have for cement paste.

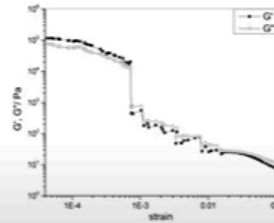
Measurement of yield stresses:

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- Stress growth experiment
- Stress/strain sweep at constant frequency



Shear stress vs time at constant angular velocity (stress growth experiment)

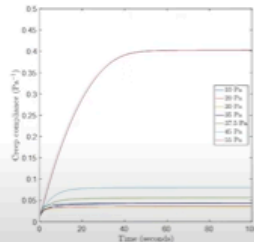


Storage modulus decreases beyond a critical strain which corresponds to the yield stress (Qian and Kawashima, 2016)

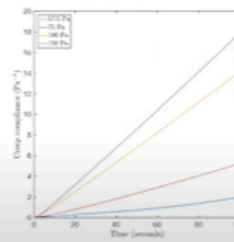
Now let us look at ways in which yield stress can be measured. We already talked about the static yield stress measurement which is also there here. You basically have a constant angular velocity, you rotate your spindle at the same speed. As we discussed earlier, your stress grows up to a certain point and then reduces and then assumes a standard value as the time increases. So you can choose this parameter that is the maximum yield stress exhibited in this stress growth experiment as your shear stress. You could also say that no, I am more interested in this value here because that is closer to my dynamic value that I want for defining my concrete properties. But generally this test is used for ascertaining the static yield stress.

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- Creep and recovery test – a direct method to determine yield stress
- The response changes from a viscoelastic solid to viscoelastic liquid like above yield stress



(a) Creep compliance below 55 Pa



(b) Creep compliance above 55 Pa

Creep response changes from a viscoelastic solid to fluid like at 55 Pa (yield stress)

But the other test is called the creep and recovery test. So here what happens is as you are loading or as you are doing the shearing, if you maintain that shearing for some time, your cement paste or cementitious material paste is going to undergo creep. And as you remove that load, there will be some recovery of the permanent strain that is or strain that is induced in your material. So creep and recovery tests are commonly used. So in this case, it is an example of creep compliance versus time.

What is creep compliance? Basically what is a creep test? You maintain a constant stress and you measure the strain with respect to time. So creep compliance which is J is strain divided by the unit stress. So creep compliance is given in terms of the strain, time dependent strain divided by the stress. So what will happen is at very low stress levels, you see here 10 Pa, 20 Pa, 30 Pa and so on, your creep compliance value is simply almost constant with time. That means your strain is not really getting built up, your paste is simply flowing. Whereas if you increase the stress to about 55 Pa, you start getting this buildup of the creep compliance with respect to time and then it assumes a steady value. So the behaviour is essentially changing at a given stress level from a viscous fluid to a viscoelastic solid and that is the yield stress, that is considered to be the yield stress in this approach. So that is one way of determining this yield stress. Of course beyond this 55 Pa, the curves are plotted here, you see an almost linear increase in your system.

Now there are other ways to do this also. In rheological experiments, we call this the study of the loss modulus and storage modulus. Again a lot of rheology involved there, I do not think we have time to really get into that level of discussion here, so I will skip that.

Issues in paste measurements:

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Issues in paste measurements



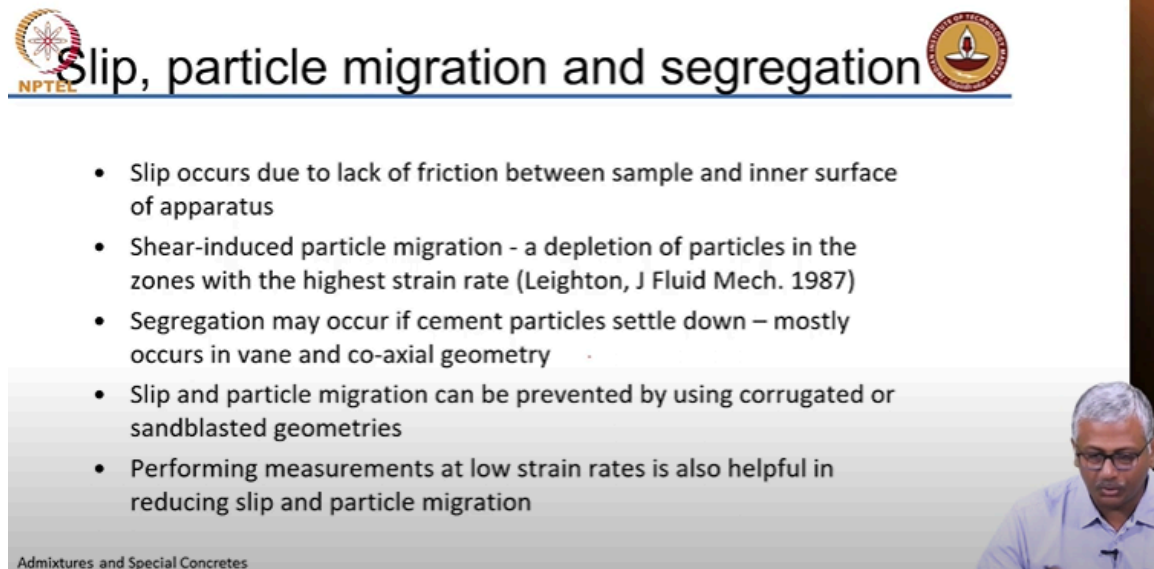
- Slip, particle migration and segregation
- Plug flow (some parts of the sample do not shear)
- Sample drying
- Transformation equations



But it is not that easy to measure rheology of cementitious systems as it is to measure the rheology of simple polymers. There are issues that can bog this study, slip, particle migration, segregation are some of the issues, plug flow, just like the instance that we talked about, some parts of the sample are not shearing, only one part of the sample is shearing. Now cement paste in the early ages, if you do not protect it from drying, it is going to start losing water and that is going to change the nature of the cement paste. And then transformation equations, how do we convert this torque and RPM to shear stress versus shear rate to get the true parameters, that is shear yield stress and plastic viscosity, how do you do that? So again there are a lot of issues, I am not again going to go into detail here, but just to make you aware that these are issues that can bog these measurements. So whenever you look at data with respect to rheology, you have to understand that it need not be entirely accurate, you need to look at the conditions at which the rheological measurement has been done and assess whether your reading has been accurate or not.

Slip, Migration and Segregation:

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The slide features a title "Slip, particle migration and segregation" in blue text, flanked by the NPTEL logo on the left and the Indian Institute of Technology (IIT) logo on the right. Below the title is a list of six bullet points. In the bottom right corner, there is a small inset photograph of a man with glasses and a light blue shirt. At the bottom left of the slide, the text "Admixtures and Special Concretes" is visible.


- Slip occurs due to lack of friction between sample and inner surface of apparatus
- Shear-induced particle migration - a depletion of particles in the zones with the highest strain rate (Leighton, J Fluid Mech. 1987)
- Segregation may occur if cement particles settle down – mostly occurs in vane and co-axial geometry
- Slip and particle migration can be prevented by using corrugated or sandblasted geometries
- Performing measurements at low strain rates is also helpful in reducing slip and particle migration

Now cement particles are in a suspended medium in the paste, with time particles may start settling down, so segregation is possible. Slip may occur if the friction is not good enough between the sample and the plate. The plate may be moving but the sample may be completely disconnected from the paste. And the problem is in cement paste you know that this will be a time dependent function, if you do initially there will be a good sticking but if you wait for some time the paste may have become cohesive or maybe there is some early shrinkage from the top surface that completely reduces the interaction between the plate and the paste. So you will have problems of slip, particle migration is


when particles start moving and you will probably not be able to have the same location of these particles during the entire test. So generally because of that it is important to perform measurements at low strain rates because all of these effects get reduced at low strain rates. Only problem is it may not exactly mimic what is happening in your actual concrete when you do the pumping or mixing strain rates are significantly high and that causes a difference in the kind of attribute that you get from these tests. The other way is to use corrugated or sandblasted geometry. So let us say in these parallel plates if you are able to have some sort of corrugations on the surface then it is able to have a better friction with your paste. That means you have to actually modify these geometries, you cannot use the same as you use for other polymers. So that is why rheological measurements in concrete become quite expensive.

Plug Flow:

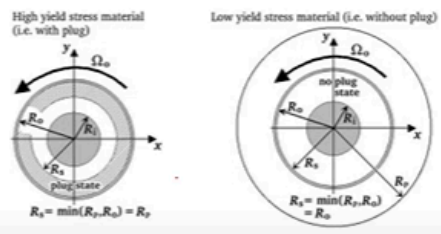
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Plug flow



- Artifacts that arise while performing measurements with yield stress fluids
- Plug flow corresponds to a situation where the sample in the rheometer is not entirely sheared
- Corrections must be applied to transformation equations to account for plug flow



Wallevik et al., 2015

Admixtures and Special Concretes

So plug flow happens when the paste basically when you are especially doing yield stress fluids like paste and concrete, the sample does not shear uniformly. A part of the sample shears whereas another part is not getting sheared. Generally you can see that if you put a vane inside the cylinder and start moving the system which is close to the blades of the vane will get sheared whereas if you are far away from the blades you probably will not have any flow at all, no shearing at all. So this is a problem of plug flow.

Sample Drying:

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Sample drying



- Occurs due to sample drying from the exposed surface
- Mainly occurs in parallel plate geometry
- Sample drying – more prone for experiments involving long time scale measurements like studying the effect of cement hydration
- Can be overcome by using a solvent trap

Admixtures and Special Concretes



Sample drying is a major problem with respect to cement paste. If your test is going to be a long term test, your sample drying or water loss from the external surface is going to create major problems. So in this case what we need to do is use what is called a solvent trap. So basically what we do is we cover the top surface with a medium that will prevent evaporation of water during the process of the testing.

Transformation equation:

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Transformation equations



- The torque – rotational velocity data obtained from the rheometer must be processed to obtain the shear stress- strain rate data
- The transformation usually automatically calculated by the rheometer software – however some of the equations used only apply to Newtonian fluids
- For co-axial geometry – accurate estimation of shear rate is difficult whereas for parallel plate – accurate estimation of shear stress becomes challenging for non-Newtonian fluids
- For cement paste – transformation procedure must be done separately – equations like Reiner–Riwlin may be used

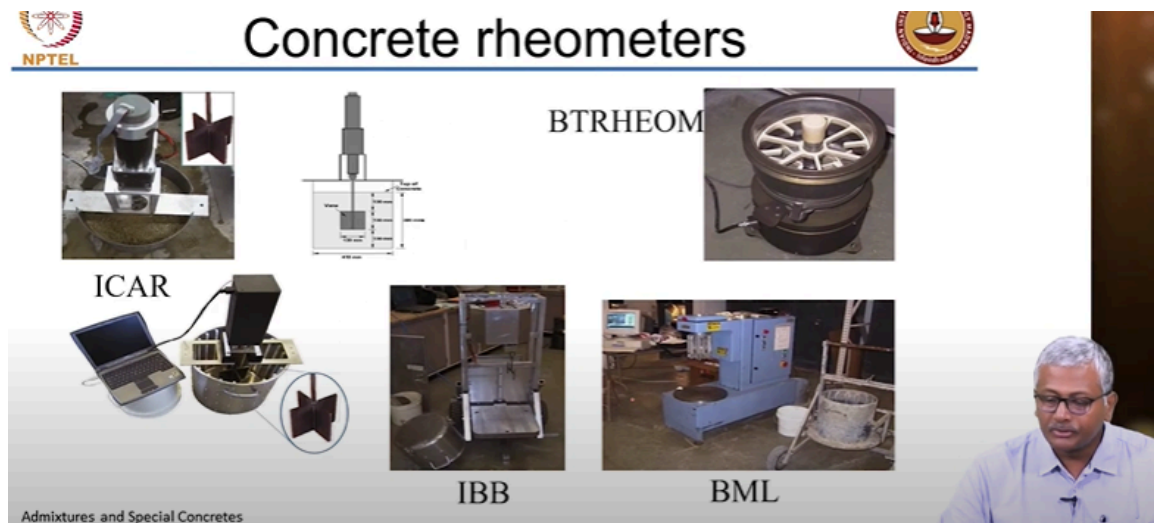
Admixtures and Special Concretes



Now again it is a little bit more complicated to understand this segment but what happens is because of the different geometries that we use, most of the equations are very clearly described for a parallel plate geometry. But when you start changing your geometries, getting the transformation equations to work equally well for cementitious materials as they do for polymeric materials, it is rather difficult. So again accurately estimating the parameters is going to be different. Of course we do not have to really get bogged down too much by that because technically we could still work with the torque values. Technically we could still work with the torque values if you are comparing different systems for instance, we can simply work with the torque values rather than trying to convert those to shear stress.

Concrete rheometers example:

(Refer to slide time: 31:15)



So just showing you some examples of concrete rheometers here, soon we will also have our concrete rheometer probably in a couple of months. So essentially the systems are quite different but they generally adopt the vane geometry. You can see the vane here which is immersed into the concrete and then you adjust the speed of rotation of the vane and you measure the torque. So as the vane is rotating at different rpm, you measure the torque and then plot the torque versus rpm.

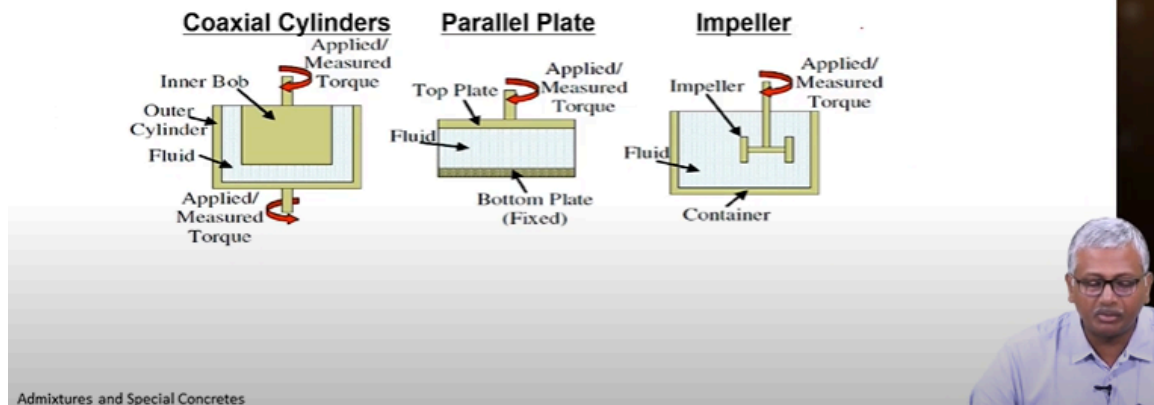
There are other geometries also, this BT rheometer has a slightly different geometry in terms of how the blades are arranged. The idea is to simply shear your concrete and measure the response of the motor while the shearing is actually happening. So people have also tried to do this by actually fitting the rheological measurement in the mixer itself. If the mixer is basically moving through the mixer blade is trying to mix the concrete, if you have some sort of a sensor on the shaft of the mixer which could then indicate the torque that could also be one way to do it.

Typical geometries:

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Typical geometries



Again this is some typical geometries. With concrete it becomes very complicated to do a coaxial cylinder and parallel plate because you really need a very good system that can actually move the entire concrete because if you have to have a gap that is 10 times the aggregate size for 20 mm aggregate we are talking about 200 mm size gap between the plates and becomes a little difficult to manage. So that is why the impeller or the vane is the best way to actually do it but there is no easy way to transform this into the actual values of shear stress and strain.