Rheology of Complex Materials
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Lecture - 60
Rheometer demonstration

So in this course on Rheology we have been at material response in terms of different classifications we looked at viscous, viscoelastic, linear viscoelastic and non-linear viscoelastic response and in these segments of lectures we are looking at some Rheometer demonstrations. In the previous segments we looked at study shear and oscillatory shear. We looked at system where there is a transition so that rheological properties change as a function of temperature. We also looked at how stress growth happens in a material and we also looked at this study shear data.

Now, in this segment we will look at another important measurement which is required as a measure of a viscoelasticity which is normal stress difference and so most of the commercial rheometers are equipped in terms of measuring the first normal stress difference. The second normal stress difference is far more difficult to measure because the measurements of pressure on the bottom plate have to be done the radial how the pressure varies as function of radius has to be done.

But as we will see now first normal stress difference is easier to measure and so we will look at a demonstration of how first normal stress difference varies for an example material and quite often of course, we have try to correlate the rheological response with the micro structure of the material. So, it becomes important to examine the material while it is being sheared. So, we will use take a look at example material where we can actually look at the structure as it is being sheared.

So, with me we have Shruthi and Jacob who are both PhD students in our group and as I mentioned last time there are several different rheometers which can do all these rheological measurements and they all will have strengths and weakness. We are doing the demonstration with the instruments which are available in our lab. So, we will first start by looking at how is normal stress difference measurement done and Jacob will demonstrate it.

Ok so now we know that when a Newtonian fluid is subjected to simple shear the normal stress difference is 0 and for and therefore for Newtonian fluid, the net force exerted in the direction normal to the direction of shear is not there. For a non Newtonian fluid the normal stresses that are developed are not negligible. Sometimes their magnitude is so high.

For example, rod climbing fluid a non Newtonian fluid which climbs the rod which is used to shear it the force I mean the fluid overcome the centrifugal force that is excited on the fluid and therefore the normal stress that is developed there in the r r direction is very high magnitude. So, normal stress that is exerted in case of a non Newtonian fluid is therefore not negligible. Therefore, it is important to also look at the normal stress data of a fluid when they subjected to simple shear.

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So, how does rheometer measure the normal stress? So, in the earlier video we you saw that the rheometer has a motor which is with using which we can measure the torque that is required for shear. But it also has a normal force sensor which is a piezoelectric transducer. This same sensor is used to detect the zeroth position of the geometry. You saw that when the geometry was brought down to the bottom plate, when the geometry touches the bottom plate the normal force shoots up and hence the zeroth position detected. And therefore the same normal force which is transducer which is used for that purpose can also be used to measure the normal stress that is developed during the course of shear flow.

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Today we are going to look at the normal stress data for 2 different types of fluids one is Newtonian fluids. I have with me 2 different Newtonian fluids one is of viscosity of 100 CP 100 centipoise, both are Silicone oil and other one is of 1000 CP. I also have with me gel which is made up of biopolymer called Pectin. Pectin is polymer that is present in the cell walls of plants. This polymer is widely used in the food industry this as an ingredient in jams and fruit juices as due to its stabilizing property, thickening properties etcetera.

So, this particular fluid during its processing stages often undergoes shear flow. And therefore, this fluid being a viscoelastic fluid will experience normal stresses which are not negligible. So, in our test today we will look at the normal stress data normal stress that is developed during the shear of shear flow of this fluid. So, what we are going to do is apply study shear flow which we saw in the earlier demo.

So, we are going to increase the shear rate from low value to high value and look at this stress shear stress developed level as well as the normal stress that is developed. So, in this rheometer whenever of thrust is exerted on to the on to the top geometry the force that is detected is positive, which means whenever a sample expanse or pushes the top geometry the force that is developed is positive value and whenever the sample shrinks or pulls the geometry towards the bottom plate with the values negative. Hence we can understand what happens during the shear flow whether sample shrinks or dilates during the course of shear.

When Jacob is saying shrinking or dilation we should remember that these are in compressible fluids. So, the effect is apparent effect. So, the fact that the it appears that this sample is pushing upword it appears as a if expanded or when the sample is pulling the over the top geometry lower than it appears as a fix contracting. But in general there is no expansion contraction in the material because it is an incompressible fluid. The other thing I just wanted to point out was also fact that silicon oils as were shown are extremely important for the overall rheological measurements. Whenever we do new material, whenever we do new type of test these standard fluids are very crucial for us to do sanity checks.

Basically to make sure that we get the results that we expect from the standard fluid ensure that the working of the instrument is according to what we expect. And that is why in this case also we are going to show you not only the results of a pectin-gel, but also the standard fluids such a silicone oil, which is supposed to be a Newtonian fluid. I am going to use a cone and plate geometry for the purpose. This is cone part with 50 mm dia with a cone angle of 1.99 degrees.

The initialization of the rheometer as well as the motor adjustment process we are all done prior to attaching the geometry. After the geometries attached the geometries detected and 0 gap is set, like it was demonstrated in the last video. Once the 0 gap is set the geometry is lifted to position where the sample can be loaded easily. Now, I am going to load this sample on to the lower plate of the rheometer and bring down the geometry to the measuring position.

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After the geometry is lowered the excess sample is trimmed out, single spatula. This graphs that if that shown here shows viscosity on the y axis and shear rate on the x axis. On the second y axis there is first normal stress difference and the test has started now. One of the important factors to remember while acquiring rheological data is the amount of time that is given to the instrument to collect one particular data point. For example, when we are doing measurements at very low strain rates it will take some time for the instrument to come to steady state.

And so therefore long time may be required and that is why here you see that the first data points have appeared, but the next set of data points will takes significant amount of time. But when we go to higher strain rates we expect the steady state to be reach faster. And so, the measurement point duration or for each and every measurement how much time is required is a very crucial decision that Rheologist has to make while doing the rheological characterization.

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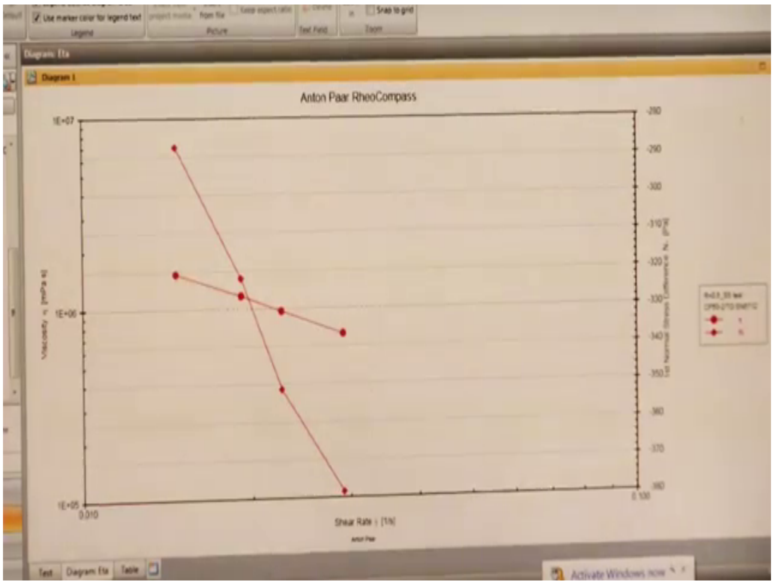

So, you can see that the first three data points have been measured and the circles or the viscosity the diamonds which are the normal stress.

Student: Difference.

Difference is also there, but you should know that the access the magnitudes are different. First of all the normal stress difference is negative.

Student: Yes.

And at least in this small range it does seem to be decreasing quite strongly, but what we will do now is to cut and show you the final data so that we can get an overall idea of how is viscosity and normal stress different for this sample.

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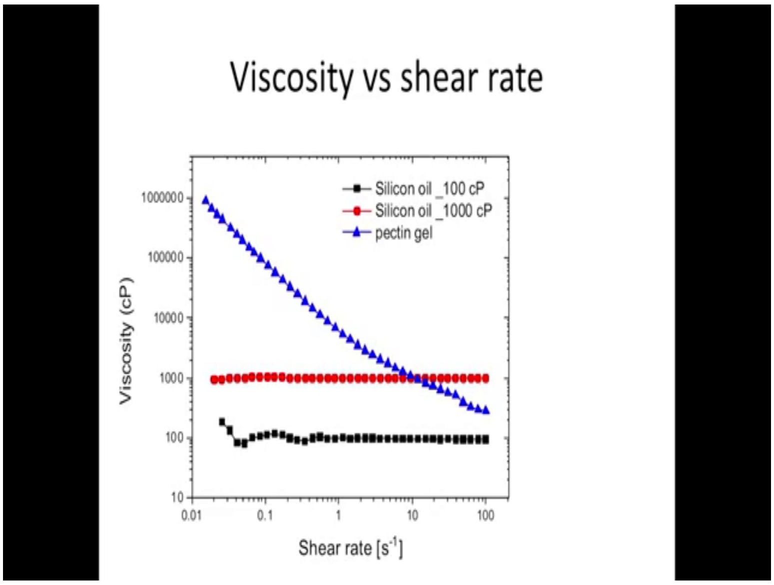

Now that we have completed the test for all three different samples, we are we can see here the results of viscosity as a function of shear rate for the three different samples that I showed you earlier. The silicon oil samples shown in black as well as red show constant value viscosity as a function of a share rate whereas, the gel pectin gel shows us strongly shear thing nature which is expected.

One thing to notice is also how the low viscosity sample, there is at low strain rate there seems to be the viscosity value not being so constant and that is again expected because at very low strain rates the torques required are extremely low and sometimes we may be in the instrument limit. So, therefore, whenever we make measurements at low strain rates we should always be careful.

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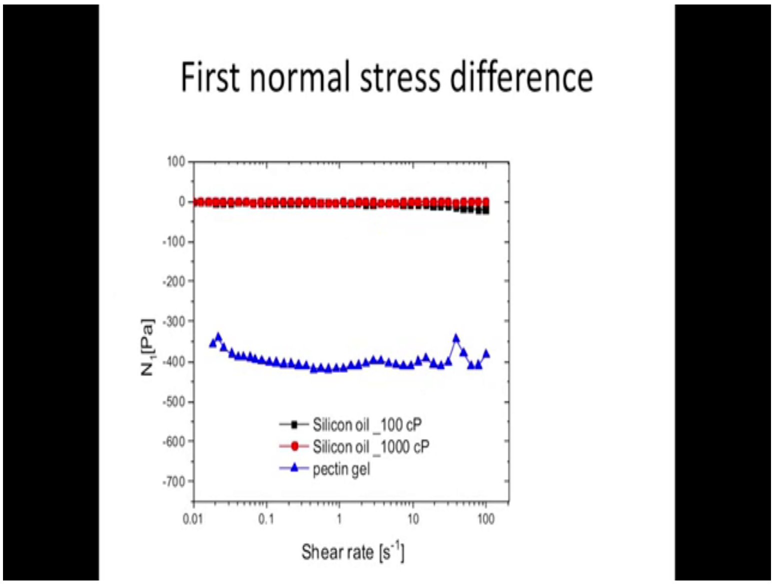

If we look at the first normal stress difference data for 3 different samples which is shown here, you can see that the Newtonian samples do not show significant value or it almost 0 value of first normal stress difference which is quite expected for a Newtonian fluid. But the gel shows large value of first normal stress difference which is also negative. To give a comparison the red curve show the first normal stress difference both in the same axis.

The value of normal stress that is developed in this case in this for this material if you when you shear it is very high compared to the shear stress. So, as I mentioned earlier the normal stress that is developed in such materials cannot be neglected because their values much high compared to the shear stress itself. So, the normal stress has a great contribution to the deformation behavior in this material.

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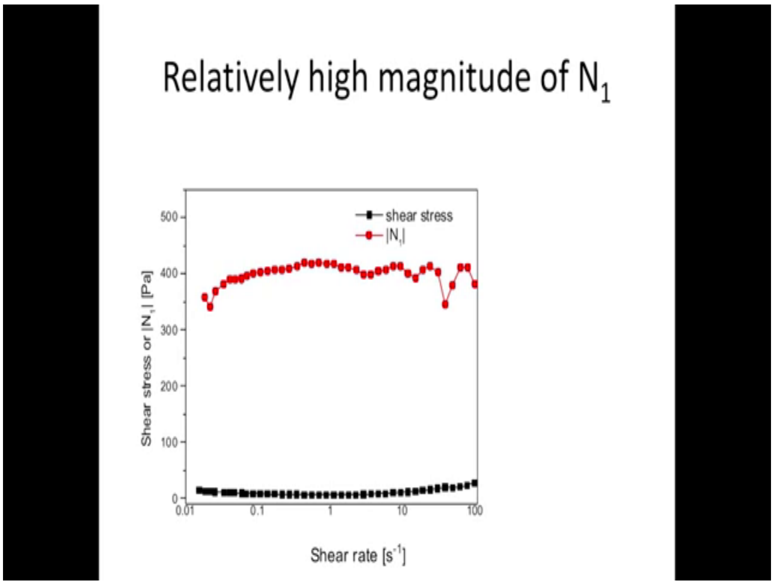

We just to remind again this is magnitude of the normal stress. As we had seen on the previous slide the in fact, it is negative normal stress. So, just to compare since we want to compare both shear and normal stresses we are plotted on the same graph. So, we are we taken just a normal stress magnitude itself. The other things to notice in this data is a fact that again at low strain and high strains. There seem to be some fluctuation in the normal stress.

So, one needs to be careful about the analyzing the data at such extreme conditions. The other interesting thing about this particular sample is the normal stress difference is almost independent of shear rate. This is not normally the case for lot of viscoelastic materials like (Refer Time: 14:40), where normal stress difference is actually a strong function of strain rate itself. So, again depending on the material and microstructure we would have different types of dependencies of normal stress difference on the strain rate.

So, in this example we saw how normal stress difference is measured and when we do the study shear measurements we can do both the shear stress measurement which is based on the torque measurement and the normal stress measurement which is based on the stress measurement. And in this particular example we saw that we the sample generates are negative first normal stress difference. And this is in fact, one of the unique materials which gives a negative normal stress difference. From now what we will see is in case we need to examine the material microstructure along with its rheology what kind of a tool we can use and specifically we will look at doing microscopy along with shear rate.

So, there are many additional attachments available with rheometer to have some additional information about the material as we do the rheology of it. If it is study shear or it is the oscillatory shear like all in all this cases what is really happening to the material we do not know. So, if you say my you say additional say attachment like microscope with this, we can find what is really happening to the material when we shear rate.

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So there are different attachment available with the with the rheometer like Rheo-SALS that is small angle light scattering and Rheo Raman microscope that is a spectrometer attach with the rheoscope rheometer and the the other one is Rheo microscopy that is the single microscope that is attach with the rheometer. So, that is what we are going to should demonstrate here. That is a simple microscope attachment rheometer. In case of, case of emulsions where the droplet sizes or the particle sizes are that are big enough to be captured with a micro symbol microscope we can go for Rheo-Microscopy.

So, here if for this demonstration we are going to use an emulsion that is an oil water emulsion water emulsion that is decane in water that is stabilized by a material particle that is silica. So, they are called Pickering emulsions.

So, as we all know emulsions have wide applications in various fields like food industries, pharmaceuticals. There are many applications with emulsions. So, there is a in all this application emulsions will be undergoing some kind of shearing happening to that. So, we should be, knowing like what is going to happen to the emulsions if it is going to be stable throughout, if it is going to break, if the droplets are going to remain as such throughout. So all these things we should be, knowing before making a product.

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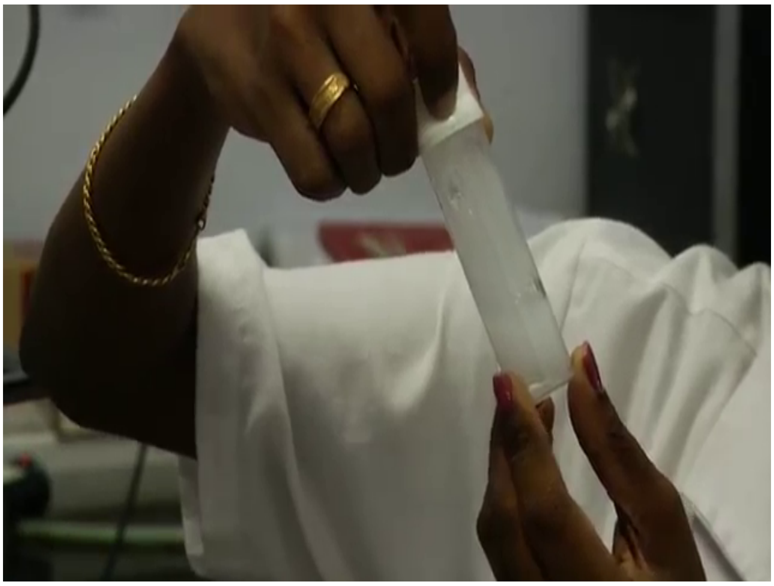

This is the material that we are going to use today. This is silica stabilized decane in water emulsion. You can see the droplets on the walls of these wires. On the microscope we can see it more clearly. Then they this an micron size droplets around 50 to 100 microns in size. Before starting the Rheo-Microscopy what we should know about the rheometer is that, we have already told that there are different kinds of filter that is available for the rheometer.

So, here what we have is a glass filter. So, it is having a glass bottom plate under which we can attach a microscope there is a microscope attached at the bottom of this. So, the, this is a stand and inside there is a microscope attached. So, it has a camera attached to that. So, there is a camera on top of which there is an objective lens of the microscope which focuses on this sample and whatever it captures will be projected on the screen.

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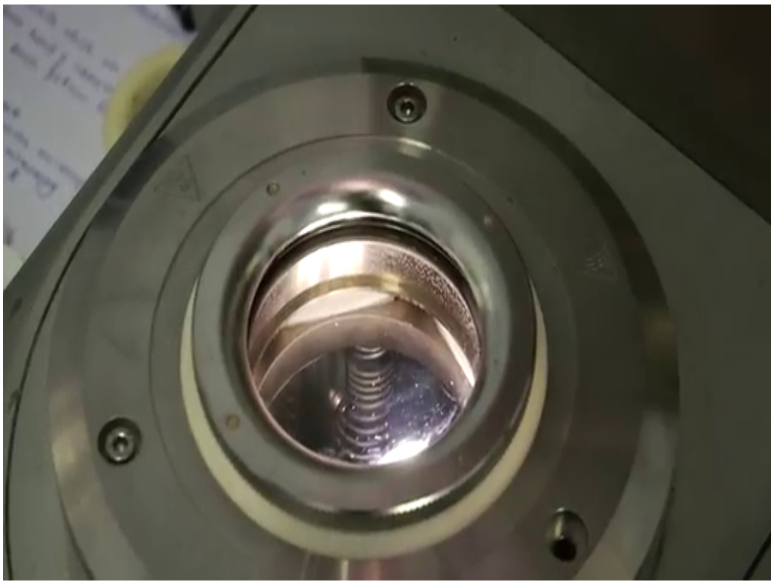

So, let us now load the sample and do a study shear test like this. Now let us load this emulsion sample and let us see what will happen to the sample as it is getting sheared. So, for Rheo-Microscopy we should need a glass filter and also we should need a glass geometry. So, the geometries which you have seen before are not transparent. Now this is a glass geometry through which we can focus on to the material and see what is happening to it. So, this is a pp 43 glass geometry that is parallel plate 43 mm, dia glass geometry. So, this is what we are going to use for this test.

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Yeah here also after attaching this glass filter I am attaching this geometry. You have to initialize the rheometer we have to do all the calibrations that is inertia adjustments, motor adjustments everything we have to do then only we should start the test. So, here I have already done all this adjustments and I am going to start the test. This xero graph is done. Now it is going to its lift position where we can load the sample. Now I am going to load this emulsion sample on to this.

Ah one of the important things in many of these samples is to make sure that the sample that you are withdrawing is actually representing the sample that you want to measure. Now many times in sample such as dispersion, emulsions basically multiphase systems it is possible that there is the small amount of settling or small amount of trimming that happens. So, before you do the measurement it is essential for you to homogenize the sample. So, that you know what you are measuring because otherwise you may be withdrawing a part of the sample and it is not re representative of the overall material that you are trying to characterize.

So, in this case also what you can see is this sample is fairly homogeneous throughout. Given that it is an emulsion. It is entirely possible that at the top there may be a small cream layer or given that it is oily water. There may be a small clear water layer below. So, in this sample you do not see such heterogeneities, but if such heterogeneities are there you need to worry about what are the measurements what part of the sample you are measuring and what do your measurements mean.

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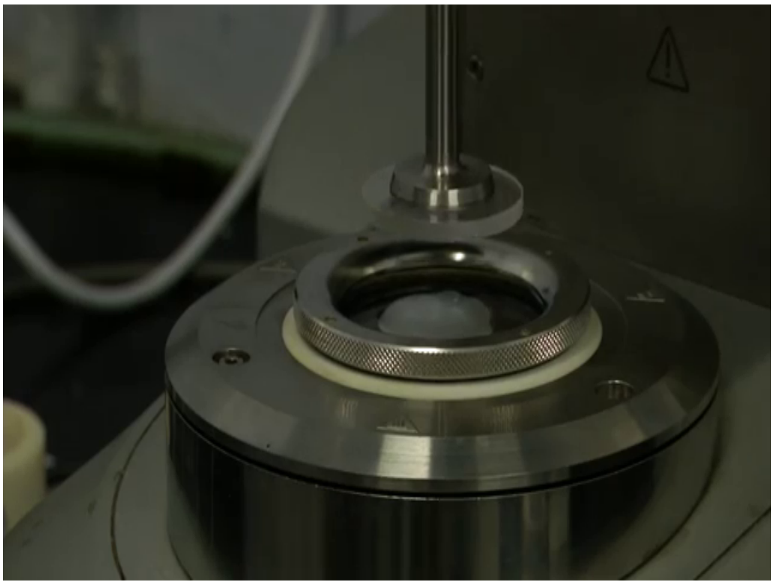

Ok now I have loaded the sample on to this glass filter. Now I will bring down the geometry to its measuring position. So, here also we have to do the trimming of the sample after loading it. But here we should little more careful because we are using a glass filter. So, we should not giving a scratch even marks on that. So, be careful by handling glass filter and glass geometries.

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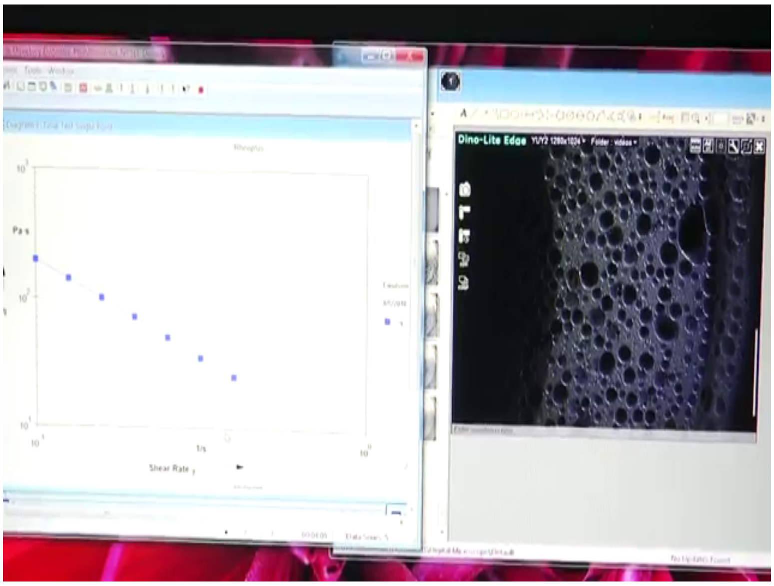

Now I have loaded the sample and this is how the sample looks like when we look it through a microscope and this is the this is the image that they microscope attach to this rheometry is capturing right now. And this is this is how the sample looks like before starting the test. Now you should notice how the drops look looks like before starting the test. This dark black colored ones are the drops droplets of oil which stabilized by silica and this is a continuous phase.

Now I am going to start the test. So, this is a steady shear test. So, here I will shear both the screens to see what is simultaneously happening to the rheology of the material as well as its micro structure when it is getting sheared. Now I will start the test. So, I am going to do a steady shear test on this with a share rate ranging from 0.1 to 1000 second inverse with a pressure of 0.1.

Ok let us start the test now. Now we can simultaneously observe what is happening to viscosity and what is happening to the material it is microstructure as it is getting shear. If you look carefully we can see that the it is moving slightly like very slowly it is move the droplets are moving very slowly because now the shear rate is only 0.1 second inverse, but when it reaches a higher shearing we can clearly see that how the droplet is moving in the continuous series ok.

So, now the test has started. The shearing is happening in this direction like in an anticlockwise direction. If quickly clearly if you carefully observe we can see that the droplets are moving in this direction. So, this is the variation viscosity as a functional shear rate there we are observing now. Even it such small shear rates we can see that this sampling showing a shear thinning behavior. Yeah it is reached almost 0.3 second inverse and we can say that some of the droplets has started coalescing and forming bigger droplets here.

ah One more important thing you can notice is some drops are in focus because they are touching the bottom plate. But there are other drops also for example, here which are maybe in the other plane. So of course, in this case this gives us an idea of the layer of the fluid which is right next to the bottom plate and hopefully whatever we are seeing is representative of what is happening to the rest of the drops because this is a uniform shear. If you recall this is simple shear where it is a constant velocity gradient so constant strain rate that is being applied. And so hopefully whatever you are measuring in terms of the droplet sizes at the bottom plate are representative of what is happening throughout the sample.

ah Another important thing to notice is in this parallel plate flow the direction is in the theta direction. So, we can see also how all the droplets are moving nicely in theta direction. So, therefore the assumption of 1-dimensional flow seems to be quit reasonably valid. All the droplets if this droplet is there it is just moving only in theta direction. Droplets are not moving in this or the other direction. Similarly a droplet which is in focus remains in focus which means the z direction motion is also not there.

Ok now the shear rate has shear rate has reached almost like 2 2 second inverse. So, now we can clearly see that many of the droplets are coalesce and formed very large droplets in this and also the droplets are moving very fast and actually the frame rate of this microscope camera which we are using is just 30 FPS.

So, after a shear rate of like 10 second inverse we may not be able to capture at the droplet motion properly. But if you say camera of a higher FPS you can capture it properly. Now the shear rate has reached almost 100 second inverse and now the speed is too high for the camera to capture. So, if we use a higher FPS camera as I said before we can capture these motions.

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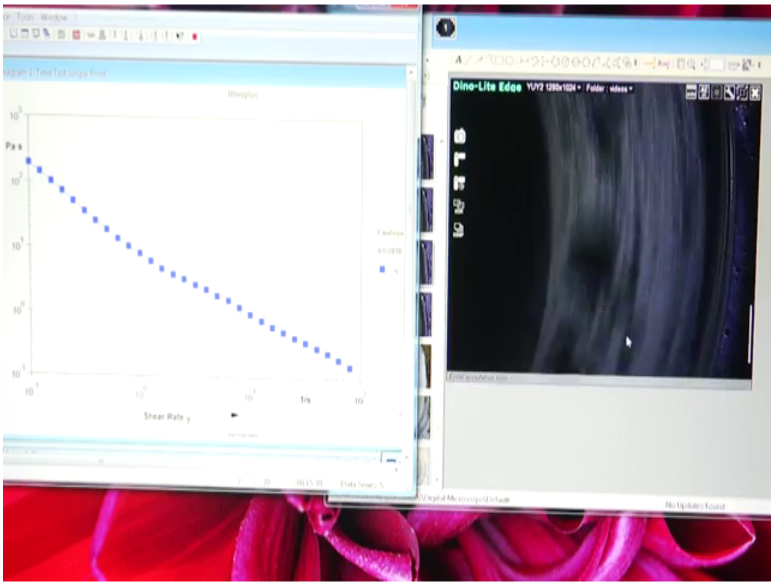

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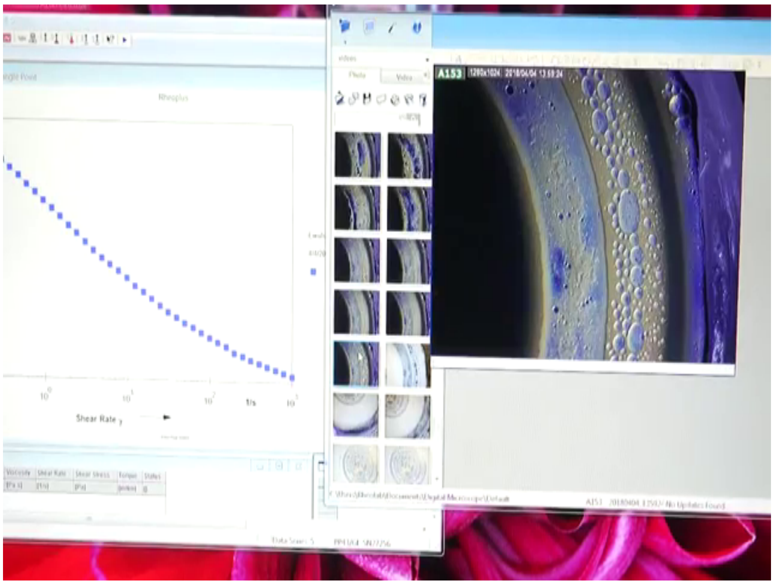

Ok now, the test is completed and we can see the clearly the shear thing in nature of this emulsion and how now the drops are arrange after the test. So, we have seen that the droplets were nicely homogeneously homogeneously arrange before starting the test and now after the test how it has arrange here we can see sorry ye. So, here we can see there is a nice arrangement of the droplets that has survive the shearing and we can see the continuous phase has separated out and formed a layer here. We have seen that at low shear rates the droplets were moving only theta direction. The arrangement after the test shows that it has some kind of secondary flows overflow in r direction to come to the periphery of the geometry. And why this emulsion droplets acquired this particular arrangement has to be actually further investigated.

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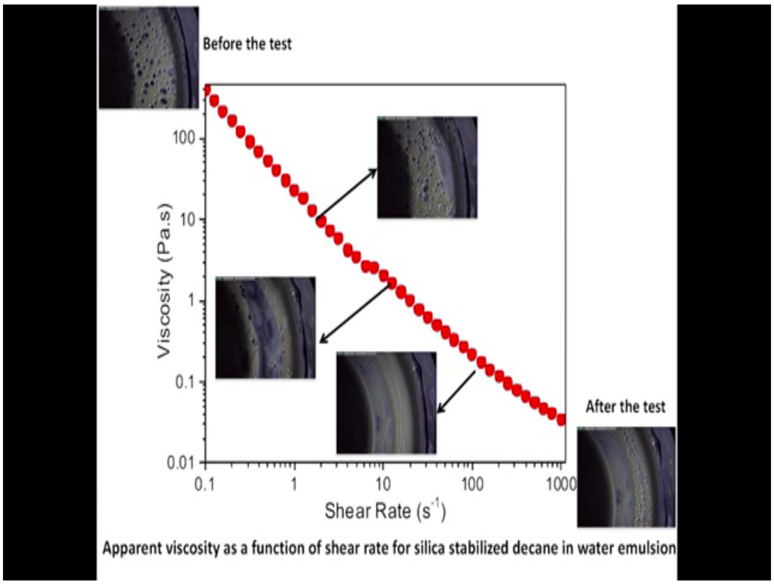

This is a summary of the test which we have carried out. We can see that before starting the test the droplets where nicely arrange like homog nicely and homogeneously arranged like this. And after the test we can see how it is arranged like this droplets which are survived has arranged along the periphery of it and the continuous phase has separated out. So when we shear the, this particular emulsion to a shear rate as highest 1000 second inverse, this is what is happening to it. And in between how this coalescence happens and how it is getting separated out, at what shear rate this happens and all are captured and it is given inside this box along with this graph.

So, this is one kind of test that we can do in Rheo-Microscopy. There are several other sets of experiments we can do, where we can focus on the whole area of the geometry and capture some kind of patterns which they are forming.