Rheology and Processing of Paints, Plastic and Elastomer based Composites

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Lecture 08

Introduction to Viscometers and Rheometers

Welcome to NPTEL online certification courses on Rheology and processing of paints, plastics and elastomer-based composites. Today we are in week 2 and this is the lecture number 2.2. And over which we will talk about the viscometers and rheometers. The concepts today covered will be first of all introduction to viscometers and viscosity and its importance, kinematic and absolute viscosity, kind of viscometers, introduction to rheometers, types of rheometers and rheological properties of polymers. Once again the keywords that you should look for while searching the content of the today's lecture.

Reynolds number is important keyword here, laminar flow, kinematic and absolute viscosity, Newtonian and non-Newtonian fluid once again although I talked about already, the shear stress and shear rate, stroke slope, terminal velocity, torque effect, strain amplitude, storage and loss modulus, oscillatory and shear, oscillatory shear and rotational shear, extensional deformations and dynamic modulus. So coming to the viscometer, the viscometer as it says is used to measure the viscosity in most circumstances. So they work for fluids whose viscosity does not change under a varying flow condition that is the mast. A fluid that can be used for a viscometer, your first postulate is that viscosity does not change under the flow conditions.

And the viscometers usually work by comparing a stationary object and a fluid flow or vice versa. Hence a viscometer could be placed in a fluid flow or move through a stationary fluid flow. So both way it is possible. So as it says the flow must have a Reynolds number in the laminar region. So it cannot be able to measure where there is a vortex or eddies form in order to record the accurate values.

So the flow condition is obviously it is below the critical Reynolds number then only it is valid. Then the measure of the resistance is taken by measuring the drag resistance during the relative motion through the fluid. And measuring these properties in different substances and materials like rubber, oil, rubber of course is a molten state or softened state, oil, plastics. So is the case of plastic it should be molten or softened depending on whether it is semi crystalline or amorphous. And then paints, coatings and adhesive etc.

Let us exemplify what we monitor basically. So examples the monitoring viscosity of

the paint what you get it. So if you have a one batch, other batch, other batch, multiple batches. If you have a viscosity measured at any instant, every instant. So if you have a consistency that means your material is consistent.

There is not much change in terms of composition or its distribution in the fluid or in the paint say for example in general. We will come back when we will talk about paint specifically. Measuring how motor oil flows when under different temperature conditions is very very important. See in the winter condition. So the viscosity is generally high.

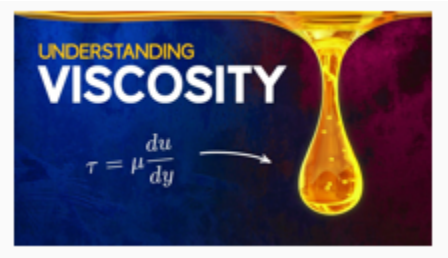
Whereas at elevated temperature in the summer the viscosity will be quite quite low. So this consistency of the viscosity is somewhat to inter fluctuation if you need to quantify it for the use, practical uses is very important. The testing the viscosity of a ink of inkjet printer. There is a classic example that we use every day. See if the viscosity is too low the fluid flow, the ink flow will be much more than the dot or matrix you want to print it for.

Or if it is too less it will be starving. So you need a particular region of range of viscosity there in a given inkjet printer it will perform, the printer will perform perfectly. The testing of rubber viscosity for maintaining the stable quality. See one of the parameters that I will exemplify further is generally used to for the quality control of the raw material raw rubber it is called Mooney viscometer. See any rubber you are getting time to time, different source to source.

So how do you have the same consistency? In that you try to have a Mooney value which is a viscometer, torque viscometer I will exemplify. So that actually is a point where you check the quality as well as the consistency, I mean not only in the raw rubber side but also the master bed. Master bed means you have rubber premixed with filler or curatives etc. Sometimes you call it filler master bed, sometimes you call it curative master bed. So those master batches whether it is scorchy or getting scorchy over time that monitoring using the viscometer like a Mooney viscometer is actually normally practiced in rubber industries.

So these are the some of the example and each of the materials whether it is a fiber, whether it is a adhesive, whether it is a paint, the viscosity first of all with that you can check the consistency. But in paints it has a different several other relevance when you apply the paint by powder coating, by spreading, by brushing. At different processing conditions viscosity also plays very very important role that we are going to elaborate later. But nonetheless hope it is clear to you what is the essence of measuring the

viscosity here to begin with. So then again going back what is viscosity technically.



Viscosity is a measure of how resistant a material is to move when you apply force to it. A material having higher viscosity has more reluctance to move. So for example if you, classic example, you just pick up a toothpaste that you all of us do in the early morning probably. So what you do? You try to push it, put the pressure and end of it with how much resistance or how easily it is coming out. Technically speaking, mathematically the viscosity is nothing but shear stress divided by shear rate.

Classically shear stress divided by shear rate. So even though by a gross definition, if shear stress is tau say for example equals to nu is into du/dy. du/dy as I already mentioned it is nothing but the shear rate. So shear stress is proportional to shear rate and the ratio is nothing but the viscosity. So normally for liquids, the conception of explaining the viscosity, the unit system is centipoise.

And of course for the solid-layer convention to always represent it as a Pascal second. So what is the relation then centipoise and Pascal second? One centipoise is equals to one millipascal second. So this is the example you should never ever forget. And this is what when I talk about the absolute viscosity. This is the unit of absolute viscosity.

I will come back. There are two different types of viscosity at least to conceptualize with. One is the absolute viscosity, another is a kinematic viscosity. I will define just now. But before that, for many liquid the stress can cause flow which is directly proportional to shear strain. The shear stress divided by shear rate is constant for a given fluid at a specific temperature.

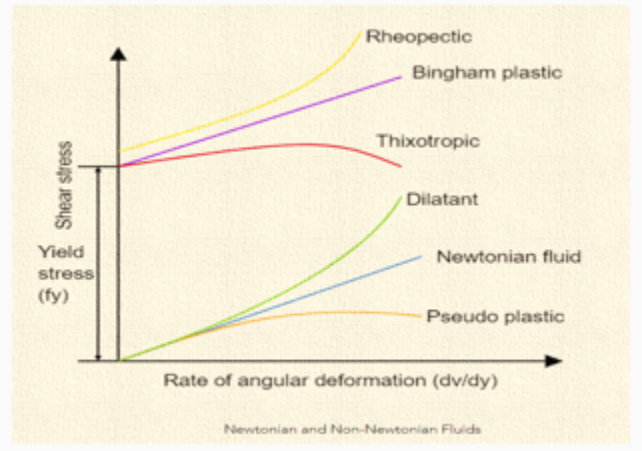
And this particular constant is called dynamic or sometimes absolute viscosity. So from this equation tau equals to as it is written here I can make it larger into gamma dot. This is the shear rate, this is the shear stress and this is the viscosity which you can either call it as a dynamic or absolute viscosity. A simple way of seeing viscosity is the thickness. Say for example, one of the classical way you have a fluid, put it fluid on a surface and

try to the thickness of it.

Obviously if the viscosity is less it will spread out at its own weight, So higher the thickness that remain over a given instant of time that means it is a more viscous material. So one of the way for rubber, one of the measurement of viscosity of rubber is a plastometer. It is based upon the same principle. I mean it is a very one of the rudimentary viscosity ever discovered by mankind. It is nothing but you take a piece of rubber, heat it at a constant temperature and try to put a specified load on that.

And at the end you measure the thickness it has. So larger the thickness remain means viscosity is high, plasticity number is high. So that way you can conceptualize the viscosity as from a layman's point of view, having without any mathematical knowledge about the viscosity as well. So with different densities, so that is what is written. A simple weight seeing viscosity is the thickness of the fluid.

But when you look at the fluids with different densities, the clearest way of describing viscosity is as resistance to the flow. So that is how it, in fact whatever I talked about with certain constant load is a flow and it is not flowing means the resistance to the flow. So once again why is measuring viscosity important? Why it is very very important to have a precise estimate of viscosity? The process conditions of a material are important both in their production as well as end use. So during the production and the end use it is very very very important. So the viscosity of a material is also useful in direct measure of its properties.



See there is a what you call as I said the modulus in a solid state. In a liquid state it is closely defined one of the parameter viscosity. And if you have some relationship, I will show you some of the relationship, the viscosity with the modulus. So if you measure viscosity, so it is indirectly you are measuring the modulus which is a physical properties. So that way your viscosity is also proportional to the molecular weight as well as density

of a material in general.

So the viscosity gives you indirect measure of the properties also not only the flow behavior of it. And thirdly it is important to measure the viscosity because not all fluids behave in the same way. So broadly fluids are classified as I mentioned Newtonian, non-Newtonian. A quick recap, again I told you already the shear stress versus shear rate or rate of angular deformation if you do it in a angular deformation way. So then you see Newtonian, it gives you a straight line pseudo plastic, in general, all the polymer melts behaves like that or shear thinning behavior.

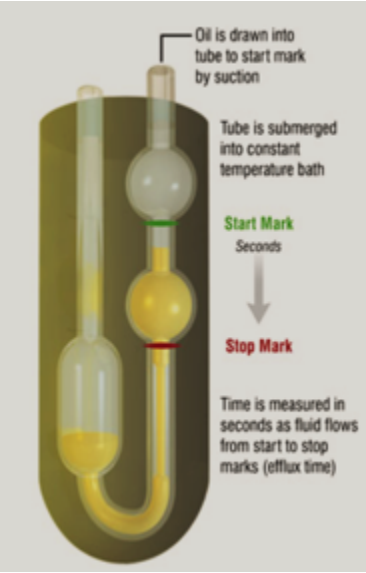
Then dilatant with shear rate the viscosity goes up and otherwise you have a Bingham fluid or rheopectic type of a fluid or thixotropic fluid in general. So these are all dividing the Newtonian fluid which is the ideal fluid, rest of the fluids are non-Newtonian in nature. So all these fluids whatever is shown here is grossly non-Newtonian fluids and what generally any polymer or any melt or any dispersion behaves like in a more of like non-Newtonian way. But it is very important now at this juncture to define kinematic and absolute viscosity as I mentioned. See kinematic viscosity again is related to the resistance to flow, resistance to go is measured by observing the fluid resistance to flow under the force of gravity.

Here absolute the force is gravity there. So under the gravity flow is happening or its deformation is happening. And absolute viscosity is measured by measuring the resistance to flow under a external or control force the tau. If you can control tau or if you can control force by area then you measure it is called the absolute viscosity. So what is the relation it holds? See as I mentioned it to you absolute viscosity has a unit of centipoise.

While in kinematic viscosity has a unit of centistokes. This is centistokes Cst means and which is nothing but I mean in the form of millimeter square per second. So actually if you look it at what is the relation between the kinematic viscosity the centistokes is nothing, but centipoise divided by specific gravity or centipoise is nothing but centipoise multiplied by specific gravity. And that way you can calculate either kinetic viscosity or absolute viscosity, kinematic viscosity or absolute viscosity and vice versa. So let us quickly try to see various types of viscometers available.

Principally, you can do it by moving a surface in touch with a fluid. You have a fluid that viscosity you want to measure and you have a moving surface and try to see the resistance, measure the resistance. Object moving through a fluid, you have a fluid you try to put a ball say for example it goes through the fluid and how much resistance it offers. Classically the Strokes law you apply that and calculate indirectly the viscosity

monitoring the velocity of the ball falling through the fluid. And then fluid flowing through the resistive component that is also another way.



So combining all this there are three ways the viscometers are designed. One is capillary or glass viscometer, rotational viscometer and falling ball or falling piston viscometer. These are the three which encompasses the three basic principles, basic methods that is in the green color suit in the left side of it. But these are the measurement techniques. By the way, before going to the viscometer and rheometer, see it grossly comes under the head of rheometry basically.

How do you measure rheological properties in that as specifying viscosity in general. So that is how you measure here. Capillary rheometer probably you are introduced from 12th standard onwards. It is nothing but there is a capillary here. And then your fluid you allow a specific volume of the fluid you allow it to flow and count on the time.

How much time it takes to flow through that capillary. Of course you may correct for any hydrodynamic effect or buoyancy effect that you have to correct it. So the Oswald viscometer is a classical one of that sort. So what is happening is a suction of flow through the force of gravity because of force of gravity it flows through. And the measurement time is then multiplied by a constant.

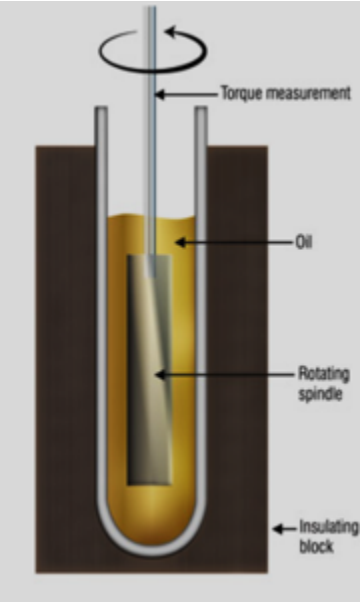
That constant varies from capillary rheometer to the capillary rheometer. But the time you calculate, time it spends for a specific volume is measured from this green to this red. This much specific volume how much time it elapse to come and come up to this fluid top layer. So that way if you measure and multiply by that constant if you know you can calculate directly the absolute viscosity. Another kinematic viscosity as I mentioned it

you just multiply with specific gravity.

So that way you can always convert it into conversion between the kinematic and absolute viscosity. See another one is very simple in design as you can see. We normally use lot of rotors in our chemical processes. It is same only thing is that you have control about the geometry of the rotor. And of course the geometry of the size of the tube is shown here.

So your yellow color thing is the oil whose viscosity we are concerned with to measure it. And then we have this spindle which can rotate. This is the torque it is exemplified and it is insulated thermally. So at a constant temperature you can fix it.

This is the typical arrangement. And the spindle is submerged with the test fluid. Test fluid is oil here. And the torque on the rotating shaft is then used to measure the fluid resistance to flow. And since the measurement does not involve the force of gravity and gravity is no more used up.

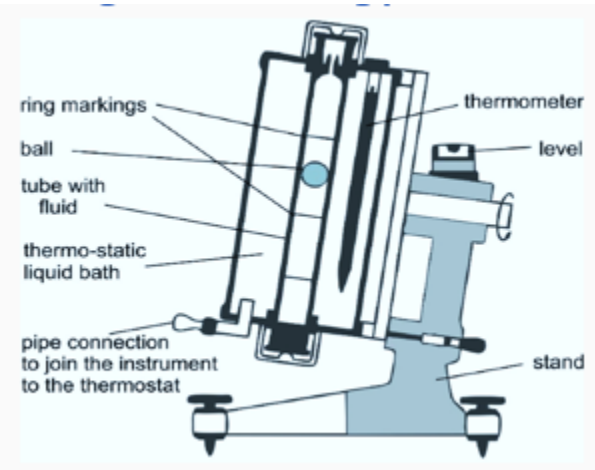


Unlike here it is flowing through gravity. In this direction g was acting. g was acting. Unlike here is the rotation. It is horizontally placed. And rather than function of the fluids internal shear stress or rotational visco I mean the rotational viscometer calculates the absolute viscosity.

No more kinematic viscosity. Tambi-seed ball. It is absolutely, absolute viscosity. Another method the third one that we talked about is the falling ball or falling piston method. As you can see from this cartoon there is a fluid body in that this ball is there.

And in a specific ring marking is a top and bottom mark.

And within that you try measuring the ball to pass through. Top to the bottom. How much time it elapses and what is the velocity of it. And its thermostat. You have a thermometer here control the temperature. Obviously all the measurement I talked about so far it has to be done at a thermostatic condition.



You need to fix up your temperature. Unlike the ball as you can see from this cartoon there is a piston. And this piston is this is the fluid and this is the continuation of course. The cylindrical tube and through which the piston will cover certain distance. And you try measuring that. So principally in this stage the ball or piston is allowed to fall into the liquid.

And the time is measured between the passing from one mark point top point and the second mark point the bottom point. So in order to calculate the viscosity in accordance with the Strokes law. The known terminal velocity, size, density of the ball piston must be known. So these are the things you should know.

And then you put it in the Strokes law standard Strokes law 6.8 RV. There you will be able to calculate here. So of course all this viscometer I talked about they have certain pros and cons. Advantages, disadvantages. See if you look it at falling ball method.

Then capillary then rotational. At least these three types I elaborated so far. See there is a range of viscosity over which it is more accurate. See that viscosity Pascal per second. That viscosity it is good for that. So capillary viscometer 10 to the power minus 4 to 10 to the power 1 capillary means Oswald type that I talked about.

And rotational 10 to the power minus 3 to 10 to the power 6 it gives you more range

wider range basically. But once again rotational viscometer is more expensive in nature. Even though the first two classes has certain drawback in terms of temperature and shear rate control. So that is very very important. These two drawbacks are there and of course the price depends on the precision you have.

And one viscometer to another viscometer there is a lot of differences in terms of prices also. So now rheometer there is a fine line of demarcation between the rheometer and viscometers. As I mentioned it to you. So far we have been talking about viscometer for a classical fluids the measure you adopts. But rheometer is an instrument that can measure the rheological properties of a polymer solution.

That is what is called rheometry. Polymer solution or melt. Using the principle of fluid rheology in which shear stresses can be measured by varying shear rate. So far so far we have been measuring and assuming viscosity to be constant. But here as I said we are coming to the non-Newtonian regime where viscosity is not a constant. It depends on the shear rate. So your measurement has to be across the shear stress has to be measured across the shear rate.

Rather than a specified at a single shear rate. That is the basic difference. So rheology is a study of deformation or flow of the matter. And technically viscosity or viscometers fall under the broader technical category of rheology. So the most broader one is your rheometry or rheology here.

And viscometry comes under that domains. Under that small definition of it. So that is how these two are different otherwise there is not much of a difference principally between the viscometry and rheometry. So rheology what you ultimately take from here. The rheological properties can play important role in the synthesis and processing of polymer solution at the industrial level. Why polymer solution? Polymer melts as well. The rheometers are ideal for measuring the viscosity of non-Newtonian fluid as I mentioned it to you.

There viscosity is no more constant. It varies with the shear rate. And they operate on the similar principle of viscometer but have a wider spread of applications. Say for example, when you take a polymer. A polymer you are doing first of all mixing with several constituents practically. And then you are allowing it to flow through a processing equipment.

Then you are curing it if it is a thermosetting type of resin. So accordingly at different level of processing different level of shear rate is involved. Or mixing say for example, when distribution of shear rate is involved at a particular bandwidth or range. So in that

context this rheometric measurement plays significant role even for your information. One single rheometer cannot capture the widest range. So many a times you have to do take make use of one rheometer another rheometer another rheometer to capture the whole range and then try to have a master curve.

May be some of the range of shear rate will remain uncovered. No man's land. In that you have to use the superposition principle to have your master curve. Master curve is nothing but viscosity as a function of shear rate from very low extreme low 10 to the power minus 6, 7 to 10 to the power minus 7. 10 to the power 6, 7, 8. So this much range you will be able to cover practically.

And then you will be able to predict the behavior at a given shear rate conditions. Say injection molding involves 10 to the power 4 second inverse shear rate at least. So this is the implication of it. So this is because non-Newtonian fluids have more complex rheological properties than Newtonian fluids. So it is a transition from viscometry to rheometries as if a transition or capturing the behavior of a Newtonian fluid to non-Newtonian fluid.

That way also you can conceptualize. So types of rheometer quickly. As I mentioned capillary rheometer for the fluid polymer solutions and capillary rheometer is as well important for non-Newtonian fluids for polymer males or solutions. And also the rotational rheometers are similar that I talked about the viscometer. Same only thing is that I am changing a fluid from Newtonian to non-Newtonian for my measurement sake. So number 1 is a capillary, number 2 is the dynamic rotational rheometer.

Third category of it. You are not allowing the complete rotation. Neither is you are allowing it to flow. Rather you are trying to put oscillatory forces and monitor its response. So oscillatory rheometer induces sine wave. Sine is very standard otherwise it can be any sort of a periodical waves. Shear deformation in the sample materials and placing them between two plates and measuring the torque effect.

And fourth one is directly torque. See you try to generate a torque and measure how much is torque changing at different temperature at different conditions say for example. And this torque rheometers are quite good monitoring the curing behavior of a material classically. So torque rheometer measures the torque or mixing screws or motors which shows how relatively hard it is to mix the sample.

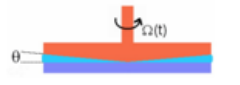
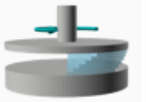
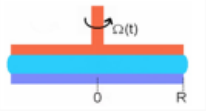
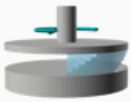
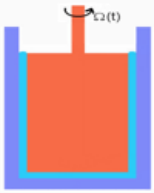
And for thermosets of course as I mentioned the curing behavior. See a capillary rheometer. I will come back again but just get used to the principles here. So there are two things. Say a capillary and through the capillary you are allowing your material to

flow here to here.

So here is a pressure. Here is a pressure at the exit. So as a result this p1 minus p2 gives you the delta p the pressure drop. And this pressure drop technically is directly proportional to the shear stress basically. So shear stress can be calculated just monitoring the pressure drop.

And second thing is the volumetric flow rate. How much volume is coming out at a given time. That is proportional to the shear rate. So that way if you monitor flow through a capillary you will be pretty well monitored the shear stress versus shear rate at different pressure drop conditions. Del p conditions. So that is the principle of it. And of course when the fluid flows through this capillary or conduit you can technically assume it is consisting of different diameter fluid layers.

Rotational rheometer

Liquid between a cone and a plate rheometer	
The liquid is sheared between an upper rotating cone and a lower fixed plate as seen from the side. The shear stress comes directly from the torque. 	The cone and plate combination evenly shears the liquid, having completely horizontal fluid layers 
Liquid between a plate and a plate rheometer	
The liquid is sheared between an upper rotating plate and a lower fixed plate, as seen from the side. The shear stress comes from the torque. 	For plate and plate, the liquid is sheared between the two plates in a controlled manner, but not evenly as with the cone-plate geometry. 
Liquid between a cup and a bob rheometer (having a cylindrical gap)	
The cross-section view below shows how the liquid shears between the central rotating bob and the fixed cup. Like the former, the shear stress comes from the torque. 	Here, the fluid layers become concentrical cylinders, such that the shear stress acts on a large surface area, compared to, e.g., the cone-plate geometry.  Pros: A cup-bob geometry is more sensitive when measuring on thin liquids compared to cone-plate or plate-plate. It is also less affected by possible evaporation effects.

And that is flowing through. And in between these fluid layers having higher and lower diameter there is some relative shear forces acting between the layers. And that is the principle. I will again come back elaborating how what is the concept of capillary

rheometer.

How it works specifically. And certain corrections of course along with that. About the rotational rheometer of course there are distinctly three varieties. One is called cone and plate. That is above here.

And it actually operates at a constant shear rate. Second variety is a parallel plate. In between parallel two plates the blue one is your fluid. And you are trying to deform it. And it gives you again the shear viscosities. But of course you do not have that control at a constant shear rate condition that apparently you get it for cone and plate.

But of course plate and plate is also important. I will come back once again there. So what is the drawback for plate and plate? It is written over here. For plate and plate the liquid shear between the two plates in a controlled manner. That shearing is very controlled.

But not evenly as for the cone and plate. So cone and plate in fact this angularity whatever you see. It is the first cone and the flat plate is to make sure the gradient of shear rate. So it nullifies basically. So ultimately end of the day it operates at a constant shear rate. Another thing in open conditions however cone and plate and plate and plate. There are chances of evaporation particularly for polymer solution which is solvent is very evaporating type of a solvent.

In that the last geometry which is called cup and bob. You have a cup inside the red one is bob and blue one is the fluid in between. And that it rotates and it measures the resistance as you can exemplify. So that is one of the big time advantage of cup and bob. It you can get rid of any kind of evaporation.

So for the paints particularly oil paints, adhesives this geometry is more preferred. I will come back when I will talk about paints and adhesive rheology. In fact technically speaking a steel also you can assume it as a fluid provided small time. So there are different types of rheometers available even to capture the fluid behavior of the steel. So different geometries is exemplified water to steel.

You see the transition apparently steel is a very high modulus solid material. So you have different different geometries those are preferred. We will come back again when we talk about rotational, I mean rheometer in a more detailed manner. So however at this juncture very low to medium, very low to high, very low to soft. And then for the solids also like steel, polymers, plastics, textiles you can still have the viscometer circle.

So another thing as I mentioned it to you is a torque geometer. So torque geometer are analytical instruments used to measure the flow and deformation characteristics of a material under different conditions. Say suppose I have to rotate this pen here. See if I rotate it in air, if I put some oil and then try to rotate, obviously the amount of torque I have to provide in is going to more and more.

And that exemplifies the viscosity is changing, getting higher and higher. And that is the principle. Even in a mixing cavity you have two rotors rotating to mix uniformity or a star is rotating. So that resistance if you have a torque meter here and measure that will give you, I mean you will be able to quantify how much resistance it is giving. And it exactly happen I mean by that principle. So the basic principle of torque geometer involves subjecting a sample of material, to control rotational forces and measuring the resulting torque or resistance to the flow.

And by monitoring the torque as a function of time, temperature and shear rate. And in fact and indeed when you process a polymer always always it is preferred you turn on your torque transducer, so that every mixing time towards a specified time you have a torque as a function of time. If it is a constant temperature, constant temperature or varying temperature, so that is the signature of that. You can always do invoke that and do post-mortem if there is something wrong happen end of the day. So applications, material characterization also, process optimization, quality control and research and development all the way through. So that is what I from the very beginning insisting that rheometer or rheometrical parameters, rheological parameters are as good as a property you measure end of the day as a testing parameter also.

Quality check parameter as well. Now the oscillatory rheometer and is a specialized instrument used to measure the rheological properties of materials particularly fluids and soft solids particularly. And as you can see the oscillatory rheometer applies control oscillatory shear forces to a sample and measures its response. And the sample is typically placed between the two parallel plates and in a cylindrical geometry as you can see from here. And by monitoring the applied stress, one plate remain stationary while the other actually oscillates.

Where you can change its amplitude and frequency. And by measuring the applied stress and the resulting strain responses of material oscillatory rheometer can determine the various rheological properties. I will come back again in details here. For rubber, particularly RPA, rubber process analyzer is one of that versatile instrument that can give you a wealth of information actually. So it is used understanding the flow behavior and structural properties and performance of materials under different conditions such as temperature, frequency and strain amplitude.

Once again why, answering why and the use of rheometers for the properties of a polymers solutions and melts. You can study the dynamic modulus. As I mentioned dynamic mechanical properties at low temperature and high temperature is similar. In one case you more invoke the term like dynamic modulus storage and loss. Other case storage and loss viscosity.

Storage and loss modulus at the low temperature and high temperature storage and loss viscosity on the contrary. The study of interacting forces in suspended and in the colloidal particles in polymer solutions. Measurement of flow behavior of polymer solution under applied stress or strain. Emulsion stability but many of the polymers are sold as emulsion and prepared as an emulsion.

Suspension of polymer solution controlling the flow of polymer solutions. Study the thermal stability of a polymer solution. Extrudability, see extrusion typically is of the order of 10 to the power 3 second inverse shear rate. Under that shear rate how is the stability of the material. Whether the structure you are making is collapsing or non-collapsing. And then processing ability of a polymer solution as well.

So at the end there are certain bibliographic as I mentioned to you already. But in addition to that I will add on the flow properties of polymer melts by Brydson. Probably I have given you earlier also. And this thing I mean got it from as a gift from Antonperz applied rheology. So that also will be a good supporting information or book for better understanding.

Quickly to conclude I talked about today about the viscometer. Definition of viscosity, kinematic vise-a-vise absolute viscosity. Types of viscometer, principle 3 capillary rotational and falling ball or falling piston. And then types of rheometer, capillary rheometer, dynamic rotational rheometer, oscillatory rheometer and torque rheometers. And their uses in polymers. In the next lecture please stay tuned as we are going to elaborate on Oswald viscometer, Brookfield viscometer, falling piston viscometer, falling ball viscometer and main flow index in a greater details. Thank you very much.