

An Overview of Measurement Techniques for Determination of Yield Stress

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ABSTRACT

Many complex fluids like food and pharmaceuticals do not flow until the applied stress exceeds a critical value, the Yield Stress. This is evident in everyday life in tasks such as squeezing toothpaste from a tube, getting ketchup out of the bottle or stirring the yoghurt in the yoghurt beaker and this is important in many industries and applications.

The determination of a yield stress as a true material constant can be difficult as the measured value can be very much dependent on the measurement technique employed and the conditions of the test, of which there are many. Consequently, there is no universal method for determining yield stress and there exist a number of approaches, which find favour across different industries and establishments.

This presentation explains and discusses the various approaches available to measure yield stress, and aspects of the practical measurement set-up and test parameters that need to be considered to obtain relevant, robust and reliable yield stress data using a rotational rheometer.

INTRODUCTION

Many complex fluids, such as network forming polymers, surfactant mesophases, emulsions etc. do not flow until the applied stress exceeds a certain critical value, known as the yield stress. Materials exhibiting this

behaviour are said to be exhibiting yield flow behaviour. The yield stress is therefore defined as the stress that must be applied to the sample before it starts to flow. Below the yield stress the sample will deform elastically (like stretching a spring), above the yield stress the sample will flow like a liquid¹.

Most fluids exhibiting a yield stress can be thought of as having a structural skeleton extending throughout the entire volume of the system. The strength of the skeleton is governed by the structure of the dispersed phase and its interactions. Normally the continuous phase is low in viscosity, however, high volume fractions of a dispersed phase and/or strong interactions between components can increase the viscosity by a thousand times or more and induce solid like behaviour at rest^{1,2}.

When solid-like complex fluids are sheared at low shear rates and below its critical strain the system is subjected to strain hardening. This is characteristic of solid-like behavior and results from elastic elements being stretched in the shear field. When such elastic elements approach their critical strain the structure begins to break down causing shear thinning (strain softening) and consequent flow. The stress at which this catastrophic breakdown of the structural skeleton occurs is the yield stress and the associated strain the yield strain.

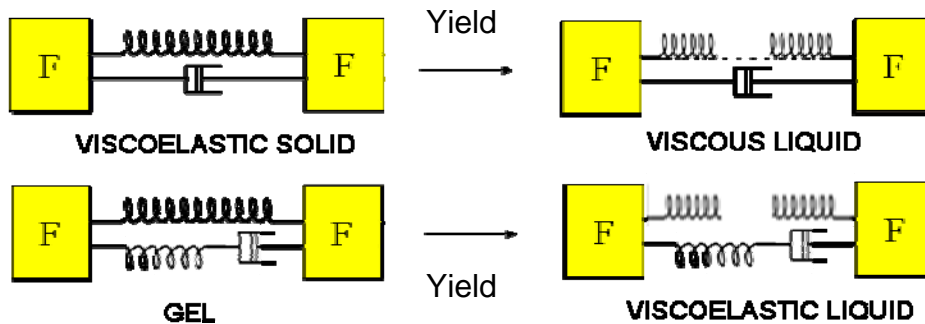


Figure 1. Illustration showing mechanical analogues and associated yielding for a viscoelastic solid and a gel.

This process can be depicted using mechanical analogues as shown in Figure 1, with a spring in parallel with a dashpot (or damper) representing a viscoelastic solid, and a spring and dashpot for a gel. In both cases the material cannot deform plastically (and flow) because it is restricted by the spring, which must first be broken. In the case of a viscoelastic solid, the yielded material will behave like a Newtonian liquid while for the gel, yielding will result in a viscoelastic liquid showing shear thinning behavior. These are simple analogues and often more complex spring/dashpot combinations would be required to describe real materials.

In the case of emulsions and foams, the solid-like behaviour results from tight or ordered packing of the dispersed phase, while in polymer gels, for example, molecular association or interaction is largely responsible.

Despite yield stress clearly being apparent in a range of daily activities such as squeezing toothpaste from a tube or dispensing Ketchup from a bottle, the concept of a true yield stress is still a topic of much debate³⁻⁸. While a glassy liquid and an entangled polymer system will behave like a solid when deformed rapidly, at longer deformation times these materials show properties of a liquid and hence do not

possess a true yield stress. According to Barnes *et al.*³ all materials can creep or flow in a similar manner on long enough timescales and consequently many materials, which are considered to have a true yield stress, are actually very high viscosity liquids. For this reason the term apparent yield stress is widely used and is considered to represent the critical stress at which there is a distinct drop in viscosity.

This key characteristic can be illustrated in Figure 2 with a material having a true yield stress showing an infinite viscosity approaching zero shear rate and a material with an apparent yield stress showing a zero shear viscosity plateau. The distinction really needs to be made at low shear rates (representing long times) since within a limited shear rate range the material may appear to have a yield stress but at much lower shear rates a zero shear viscosity plateau can be observed. It is, however, important when making such distinction that measurement artifacts such as wall slip, thixotropy and instrument resolution are accounted for.

While the concept of an apparent yield stress or critical stress as opposed to a true yield stress is valid for many materials, there is strong evidence to suggest that this is not the case for all materials⁴⁻⁶.

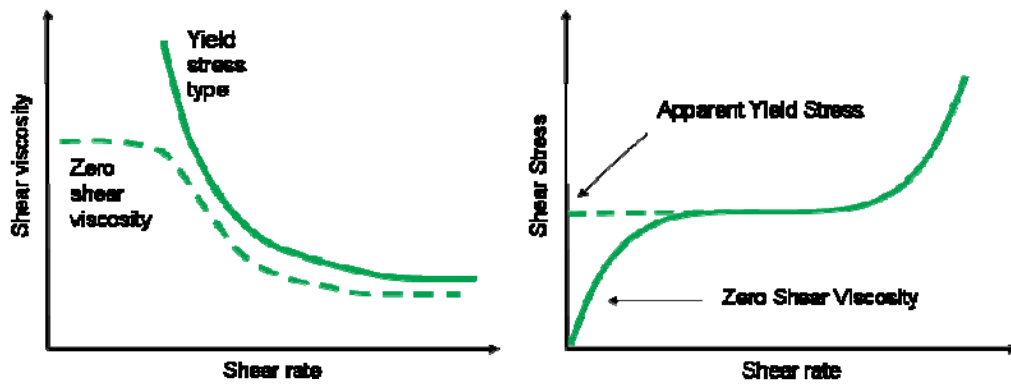


Figure 2. Illustration showing an expected flow curve for a material with a true yield stress and a zero shear viscosity (left) and a material which appears to have a yield stress but shows viscous behaviour at much lower shear rates (right).

What does this matter? For short time processes such as pumping, stirring and extrusion not that much, however, for longer-term processes such as those impacted by gravity, sedimentation for example, and then establishing the presence of a true yield stress can be important⁹.

The determination of a yield stress as a true material constant can be difficult as the measured value can be very much dependent on the measurement technique employed and the conditions of the test. Consequently, there is no universal method for determining yield stress and there exist a number of approaches, which find favors across different industries and establishments^{10,11}.

One such variable, which can affect the measured yield stress value, is time. Many complex fluids are thixotropic in nature and can change structurally with time of applied shear and/or take a finite time to recover after yielding. This can be particularly important when loading samples prior to measurement since this process often requires yielding the material first^{12, 13}. Timescale of testing is important since complex fluids generally undergo stress relaxation when exposed to an applied strain. This occurs through micro-structural rearrangement, which can ultimately influence the network structure and hence the yield stress⁸.

Micro-structural relaxation processes are best evaluated using dynamic testing on a rotational rheometer with inverse frequency correlating with time. Some typical frequency spectrums and their mechanical analogues are shown in Figure 3. Since G' is the modulus associated with elasticity (and connectivity) then when this value exceeds the viscous modulus (G''), which is associated with flow, the material can be considered to have a network structure and hence a yield stress. For a material to have a true yield stress then G' must exceed G'' at infinitely low frequencies, which would be the case for a viscoelastic solid and an ideal gel. For a viscoelastic liquid the material will only appear to yield in the frequency range where G' exceeds G'' and thus these materials can be considered to have an apparent yield stress or critical stress.

Another important factor is temperature. At higher temperatures, material components have more thermal energy and hence a lower stress input is required to initiate flow. Consequently, yield stress tends to decrease with increasing temperature so long as there is no thermally induced structural enhancement at elevated temperatures¹.

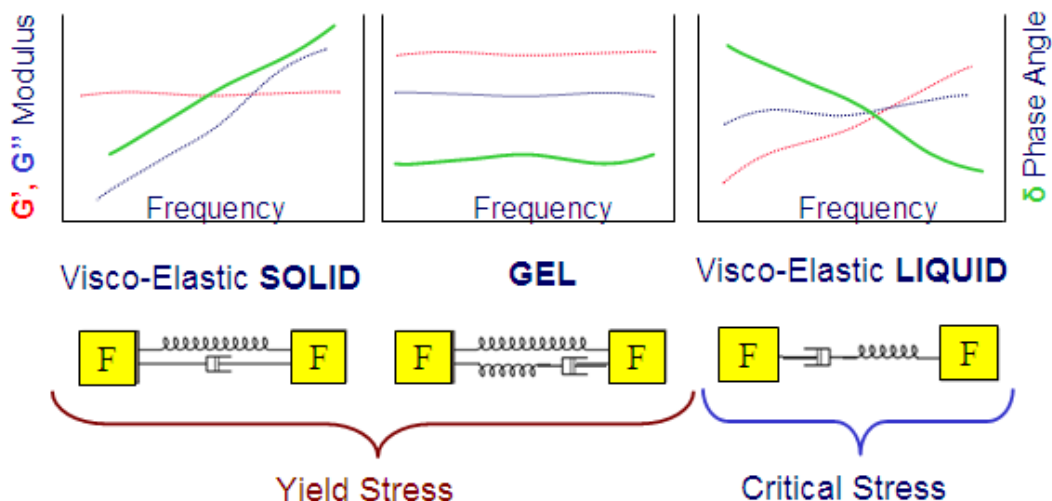


Figure 3. Illustration showing some typical frequency profiles for materials with a yield stress/critical stress and their mechanical analogues.

There are various methods for measuring the yield stress of a material ranging from accurate rheometric techniques to some cruder non-absolute techniques. Some examples of non-absolute tests used in industry include the slump test, which involves measuring the degree of slumping of a cylindrical volume of material on a horizontal plane¹³, and the Inclined Plane Test, which involves measuring the equilibrium sample thickness on an inclined surface following an initial, period of flow¹⁵.

For the purpose of this paper, the focus will be on techniques available for use with a rotational rheometer, of which there are several available methods including creep, stress ramp, stress growth, oscillatory techniques and model fitting. A detailed summary of each technique will be given along with some discussion regarding best practice for making measurements and how to avoid measurement artefacts particularly regarding wall slip.

YIELD STRESS METHODS

Model Fitting

The traditional method for measuring yield stress on a rotational rheometer or

viscometer was by fitting models to the measured rheograms (shear stress versus shear rate data) and extrapolating to zero shear rate². The simplest of these models is the Bingham model, which is often used to describe the behavior of concentrated suspensions of solid particles in Newtonian liquids. These materials often show an apparent yield stress followed by nearly Newtonian flow above the yield stress. The Bingham model can be written mathematically as:

$$\sigma = \sigma_0 + \eta_B \dot{\gamma} \quad (1)$$

where σ_0 is the yield stress and η_B is the Bingham viscosity or plastic viscosity. It should be noted that the Bingham viscosity is not a real viscosity value; it just describes the slope of the Newtonian portion of the curve.

An alternative model to the Bingham model is the Casson model. This model has all components in the Bingham equation raised to the power of 0.5, and consequently has a more gradual transition between the yield and Newtonian regions. It tends to fit many materials better than the Bingham model and is widely used to characterize inks and chocolate in particular.

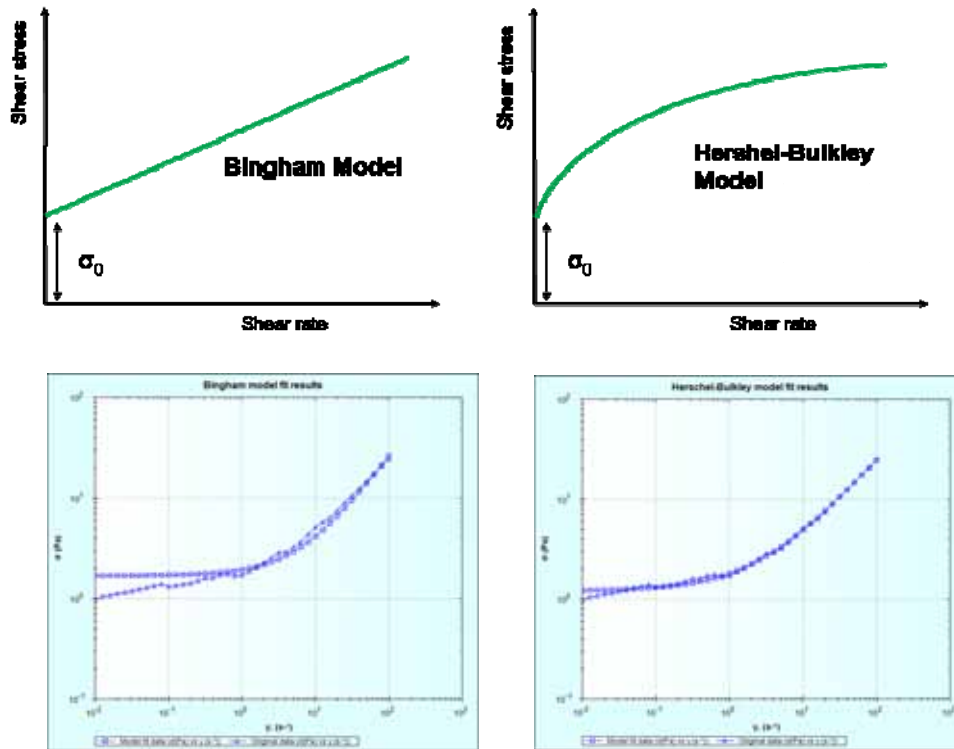


Figure 4. Illustration of Bingham and Herschel-Bulkley model fits using linear scaling on the top and using logarithmic scaling below using mayonnaise measurement data.

The Casson equation can be written as,

$$\sqrt{\sigma} = \sqrt{\sigma_0} + \sqrt{\eta_c \dot{\gamma}} \quad (2)$$

where σ_0 is the yield stress and η_c is the Casson viscosity, which relates to the high shear rate viscosity. Another yield stress model is the Herschel-Bulkley model. Unlike the Bingham equation, this model describes non-Newtonian behavior after yielding and is basically a power law model with a yield stress term. The Herschel-Bulkley equation is written as follows;

$$\sigma = \sigma_0 + K \dot{\gamma}^n \quad (3)$$

where K is the consistency and n is the shear thinning index. The latter term describes the degree to which a material is shear thinning

($n < 1$) or shear thickening ($n > 1$).

Stress-shear rate curves for a Herschel-Bulkley and Bingham type fluid are illustrated in Figure 4. Note these are presented on linear scaling but will have different profiles when displayed logarithmically, which is how such curves are usually represented.

To determine which model is most appropriate it is necessary to measure the steady shear stress over a range of shear rates and fit each model to the data. The correlation coefficient is then a good indicator of the goodness of fit. The range of data used in the analysis can, however, have a bearing on the results obtained since one model might better fit the low shear data and another the high shear data.

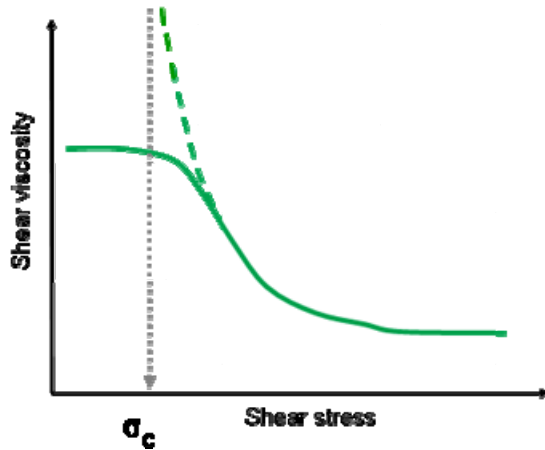


Figure 5. Illustration showing an Ellis model fitted to the flow curve of a shear thinning liquid.

It should be noted that yield stress values determined by model fitting are often termed dynamic yield stresses as opposed to the static yield stress attributed to other methods such as stress ramps and stress growth. The dynamic yield stress is defined as the minimum stress required for maintaining flow, while the static yield stress is defined as the stress required for initiating flow and is often higher in value. It is usually better to measure the static yield stress when looking at initiating flow in a material i.e. pumping, while dynamic yield stress may be more applicable in applications for maintaining or stopping flow after initiation.

There are additional models that can be used to estimate the yield stress, or more appropriately, the critical shear stress for materials having a zero shear viscosity. These additional models are modified versions of the Ellis and Cross models for viscosity versus shear stress and viscosity versus shear rate data respectively. The Ellis model can be written as follows.

$$\eta = \frac{\eta_0 - \eta_\infty}{1 + \left[\frac{\sigma}{\sigma_c} \right]^m} \quad (4)$$

Where η is the viscosity, η_0 is the zero shear viscosity, σ is the stress and σ_c is the critical shear stress. The critical shear stress is the stress at which the onset of non-linearity occurs and is essentially the asymptotic value of the shear stress at infinite viscosity assuming power law behaviour. The exponent m is a shear thinning index, which is a measure of the degree of non-linearity, and η_∞ is the infinite shear viscosity (limiting value of viscosity at very high shear rates)

Stress Ramp

One of the quickest and easiest methods for measuring yield stress on a stress controlled rheometer is to perform a shear stress ramp and determine the stress at which a viscosity peak is observed (Figure 6). Prior to this viscosity peak, the material is undergoing elastic deformation and hence the strain rate is almost constant even though the stress is increasing linearly. This peak in viscosity represents the point at which the elastic structure breaks down.

Since yield stress can be a time dependent property then the stress ramp rate can be an important factor. Therefore, it is essential to use a standard or constant value when comparing between samples.

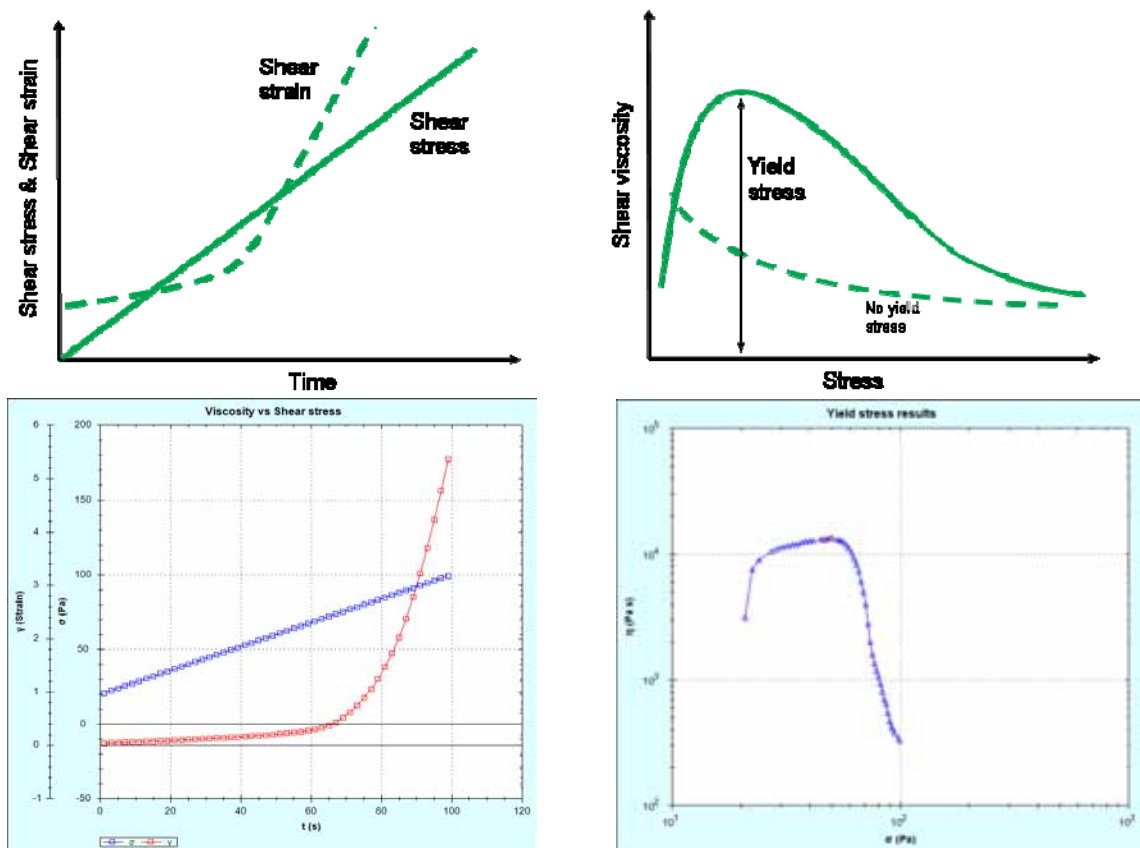


Figure 6. Shear stress-strain curve (left) and corresponding viscosity-stress curve (right) and below are shown similar curves for mayonnaise.

Stress Growth

With a stress ramp test, a constantly increasing stress is applied and the resultant strain rate or shear rate monitored with time. Conversely, a stress growth test involves applying a constantly increasing strain (constant shear rate) and monitoring the stress build-up with time. Below its critical strain, the sample is subjected to work hardening resulting from elastic elements being stretched in the shear field. When such elastic elements approach their critical strain the structure begins to break down causing shear thinning (strain softening) and subsequent flow. This event coincides with a peak value in shear stress, which is equal to the yield stress, before leveling off to its

equilibrium value. This is depicted diagrammatically in Figure 7.

Usually a low shear rate is employed in these tests to account for time relaxation properties of the material, although different shear rates can be employed depending on the application of interest. Fast processes such as dispensing occur on short timescales, and thus correspond with higher shear rates, while stability to sedimentation/creaming occurs over longer times and is better evaluated at lower shear rates. Since yield stress is generally a time dependent property then the measured values can be different. A shear rate of 0.01s^{-1} , however, is commonly used and has been found to give good agreement with other yield stress methods¹⁷.

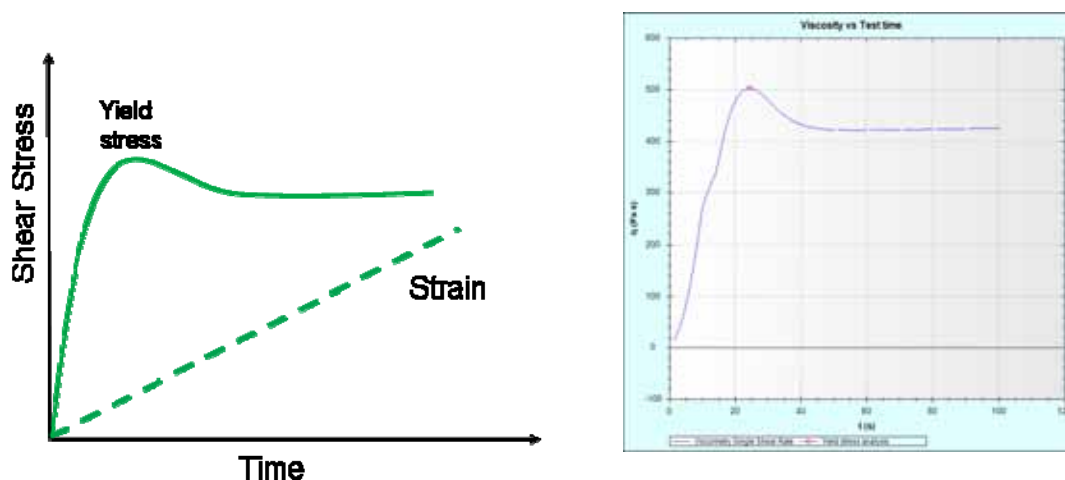


Figure 7. Illustration showing the stress evolution of a yield stress material at constant shear rate to the left and real data for a pharmaceutical gel at a shear rate of 0.01s^{-1} .

Oscillation Amplitude Sweep

This test involves applying an increasing oscillatory stress or strain and monitoring the corresponding changes in the elastic modulus (G'), or the elastic stress (σ') with increasing amplitude. There are different ways of interpreting yield stress from an amplitude sweep, as shown diagrammatically in Figure 8. Some consider the initial drop in G' a measure of the yield point since this represents the onset of non-linearity and hence structural breakdown, while others consider the G'/G'' cross-over to be the yield point since this represents the transition from solid to liquid like behaviour¹⁶. The zone spanning these two events is often referred to as the yield zone since it represents the transition from solid to liquid like behaviour.

A more recent method for determining the yield stress by means of oscillatory testing involves the measurement of the elastic stress component (σ'), which is associated with the elastic modulus (G'), as a function of strain amplitude^{17, 18}. The yield stress is taken to be the peak value of the elastic stress, and the corresponding

strain value the yield strain. This stress value generally is somewhere between the values corresponding to the G'/G'' crossover and the initial drop in G' . It has been shown to give a more reliable measurement of the yield stress and correlates well with other methods^{17, 18}.

It is important to note that test frequency can influence the measured yield stress depending on the relaxation behaviour of the material under test. Since G' generally decreases with decreasing frequency for these types of material then yield stress tends to follow a similar trend. While lower frequencies will give a better indication of materials properties at rest, performing an amplitude sweep at such low frequencies can greatly increase the time of the test. Consequently, values between 0.01 and 10 Hz are commonly employed depending on the application of interest. Care should be taken when using serrated parallel plate for example since the geometry is oscillating around a fixed position and can potentially leave voids in the material¹⁹.

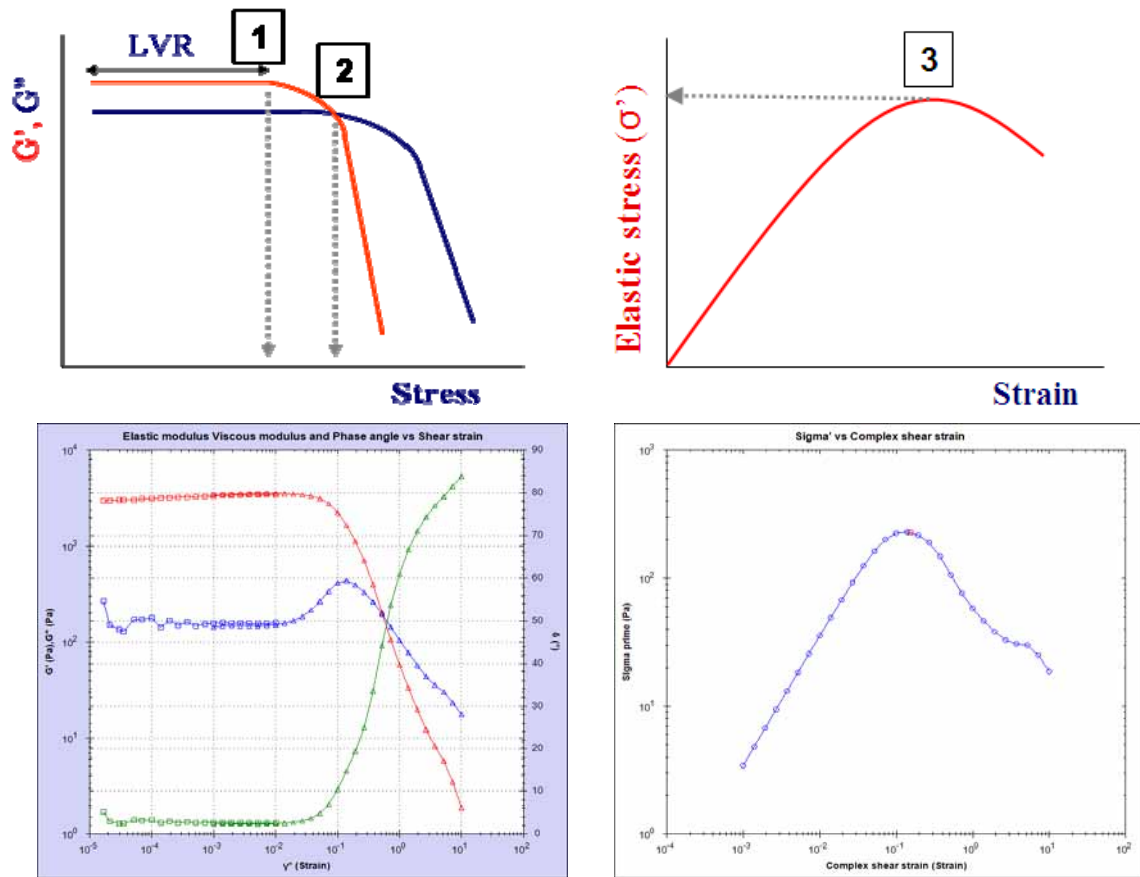


Figure 8. Illustration showing points commonly used to determine the yield stress and strain from an oscillation amplitude sweep and below are data from measurements using cosmetically grade of laponite.

Multiple Creep

What is considered one of the most accurate methods for determining yield stress is a multiple creep test. This involves performing a series of creep tests using different applied stresses and looking for changes in the gradient of the compliance versus time curve. Depending upon the nature of the material being tested, the response can be quite different as illustrated in Figure 9.

Since the actual change of strain will be dependent upon the applied stress, it is usual to talk about the compliance rather than the strain. The creep shear compliance (J) can be determined from the preset shear stress (σ) and the resulting deformation (γ) through:

$$J(t) = \gamma(t) / \sigma_0 \quad (5)$$

Using this notion, creep curves generated at different stresses can be directly compared. All $J(t)$ curves overlap with each other independent of the applied stress as long as the stress is within the linear viscoelastic region and below its critical strain and critical stress. When this criterion is no longer met, the material is considered to have yielded. From Figure 10 it can be deduced that the material under test has a yield stress between 3 and 4 Pa, since at 4Pa the curve no longer overlays the lower stress data. To achieve a more precise estimate of the yield stress it would be necessary to repeat the test with small incremental increases in stress between these two values.

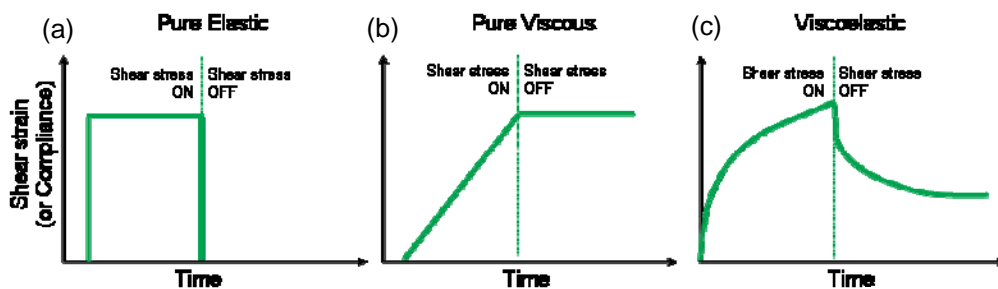


Figure 9. Illustration showing the strain response to an applied stress for (a) purely elastic material (b) purely viscous material (c) viscoelastic material.

Again test time can be important as also depicted in Figure 10, where yielding occurs only after a given time at 4Pa and yet instantaneously at 5Pa. This again is because many materials are structurally dynamic.

Generally a test time ranging from 2-10 minutes is usually sufficient for such a test but longer times can be required if the material relaxes over a longer period. Allowing the material to relax between creep tests is important also since otherwise stresses will be accumulated in the sample giving artificially low values for the yield stress. This can be achieved by employing a relaxation step between creep tests, which involves applying a zero stress for a time at least equivalent to the creep time.

Tangent Analysis

Tangent analysis is another common method for determining yield stress, which

can be used in both oscillatory and steady shear techniques. In oscillatory tests if a single tangent is applied to the linear region of the curve then the yield stress is often taken as the stress at which the curve begins to deviate from this tangent. This is essentially the end of the linear region, which is labeled 1 in Figure 8. The more common use of tangent analysis is to apply tangents to the linear viscoelastic region and the flow region, with the yield stress being the stress value at which the two tangents cross. Most often tangents are fitted to the stress-strain data as opposed to modulus-strain data, although the values should be equivalent. This same treatment can be applied to a steady shear stress ramp also, as shown in Figure 11(a). It should be noted that when fitting tangents for determination of yield stress, the data should be plotted logarithmically since most yielding materials demonstrate power law behavior.

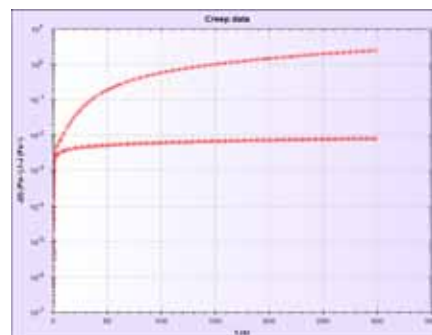
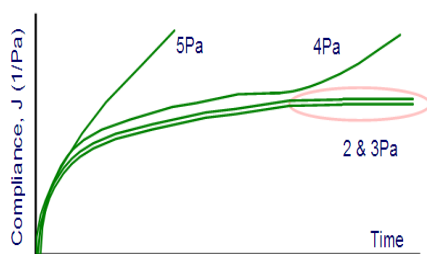


Figure 10. Illustration showing a multiple creep test with yielding at 4 Pa and data from a commercial scalping crème with a yield stress of 12 Pa.

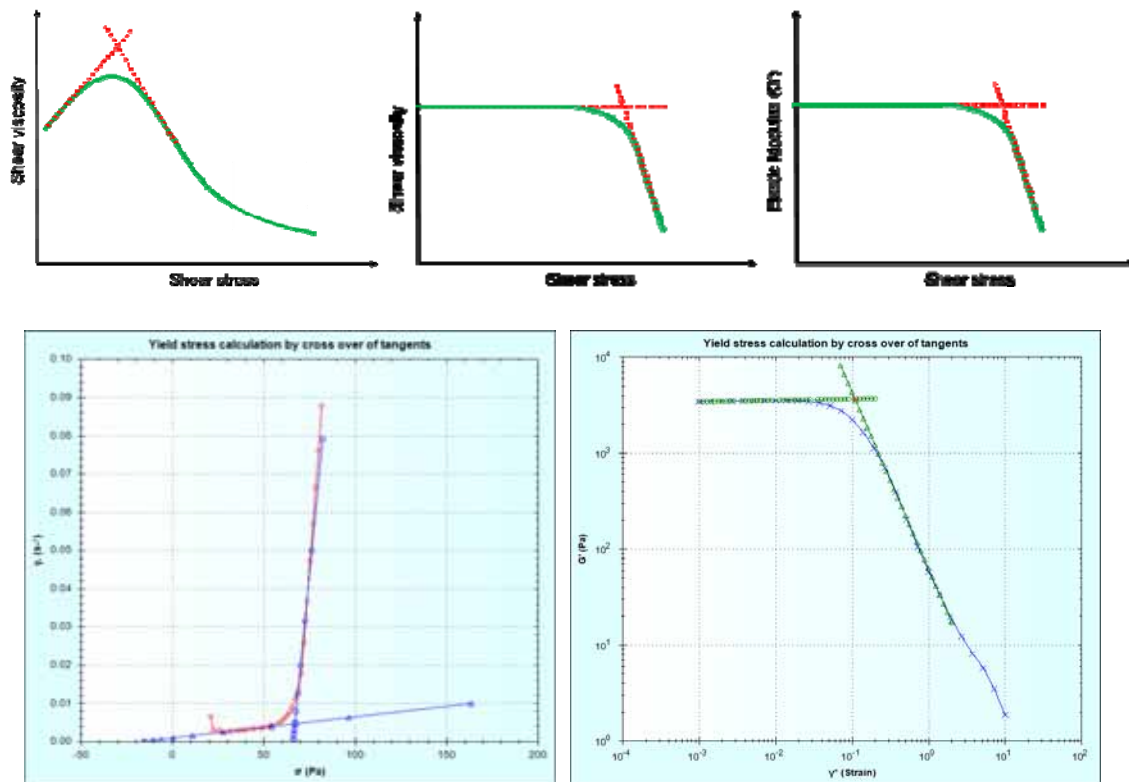


Figure 11. Illustration showing yield stress/critical stress determination by tangent analysis using steady shear testing (a and b) and oscillation testing (c). Below is the tangential analysis of data from a mayonnaise in a linear ramp in shear stress (here plotted as shear rate vs. shear stress) to the left and to the right is the tangential analysis of the laponite sample (plotted as G' vs. strain).

MEASUREMENT SYSTEMS

The choice of measuring system is important when making any rheological measurement and particularly when measuring yield stress. When making shear rheological measurements on structured liquids; in particular suspensions, emulsions or foams, there is a high probability that the measurement may be affected by a phenomenon known as 'wall slip'. Wall slip generally results from a local depletion of the dispersed phase near the geometry wall, which effectively forms a lubrication layer at the surface. Consequently, bulk rheological properties are no longer being accurately measured leading to an underestimation of the true viscosity and the yield stress. A similar effect can be observed

when measuring stiff solid like materials where there is insufficient friction between the sample and the wall to support the applied stress^{18, 21, 22}.

Wall slip can be counteracted in a number of ways, most notably by use of roughened or serrated geometries, which effectively take the geometry motion into the bulk of the liquid, maximizing sample-sample contact at the expense of sample-wall interactions. The degree of roughness generally depends on the stiffness of the material and the size of any dispersed matter, with serrated plates usually preferred for stiffer samples and large particles. Figure 8 shows the consequence of wall slip for a concentrated particle suspension, as measured using smooth parallel plates.

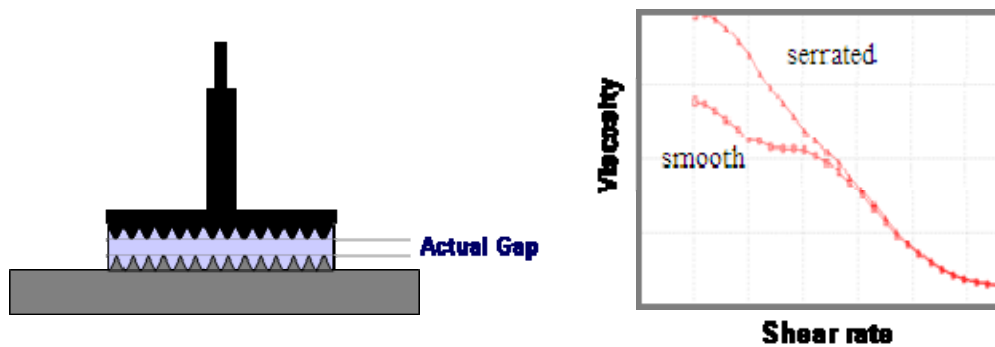


Figure 12. Illustration of serrated parallel plates used to minimize slip and associated data for a the flow curve of a dispersion measured with smooth and serrated plates.

The apparent ‘dog leg’ in the flow curve is a familiar characteristic of wall slip, which in this case is largely eliminated by the use of serrated plates.

When working with a plate set-up it is often preferable to use a cone measuring system. This is because the shear stress is essentially the same over the entire cone surface and hence the material should yield homogeneously across the sample radius. In the case of a parallel plate, the measured stress is a function of the applied shear rate, which varies with radius. Hence, sample exposed to the outer radius of the plate will yield before that in the central zone and consequently in some tests a plate may give slightly different results. Roughened cones are available to minimize the effects of slip, however, if the sample contains large particles and/or requires a serrated system due to extensive slippage then a parallel plate may be the only means of proper measurement using a plate set-up. The gap employed in such tests can also be important since slip effects are usually more prevalent at small gaps. This is because slip velocity at the plate surface is in addition to the geometry velocity, which gets smaller relative to the constant slip velocity as the gap gets smaller¹. When working with some materials, particularly paste like materials, there may be limitations on the working gap

that can be used to obtain moving at different speeds and a characteristic inflection on the shear stress-shear rate curve.

Working with a cylinder set-up, vanes and splined geometries can be used to minimize slip and work in a similar way to the serrated plate systems. The former is accurate yield stress measurements. This is because such materials can show inhomogeneous flow behavior when larger gaps are employed and can cause partial yielding or fracture across the gap. This is usually visually evident, with close examination revealing two distinct layers often recommended for concentrated dispersions and emulsions, which are prone to slippage since this maximizes sample-sample contact²⁰⁻²². Another benefit of the vane tool is that it can be inserted into the sample with little disturbance to the structure. This can be important since many complex fluids are thixotropic and may take a finite time to recover their structure after loading or in some cases not at all. The vane can also potentially be used with the product in its original container, dimensions permitting, which mean there is no need to transfer the sample to a measurement cup, which again prevents structural damage prior to measurement.

$$\sigma = M / 2\pi R^2 L$$

$$R_0 > R_1 (\sigma_{YB} / \sigma_{YW})^{1/2}$$

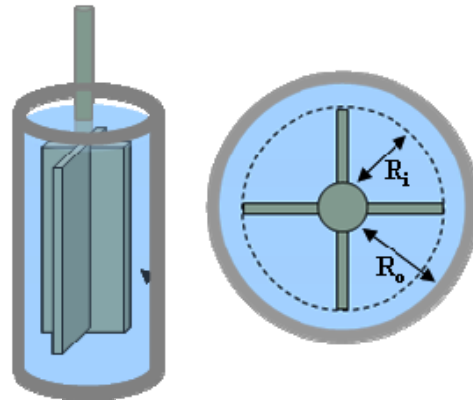


Figure 13. Illustration of vane tool in a smooth cup and associated stress equation. M is the torque and L the vane length; σ_{YB} is bulk yield stress and σ_{YW} the wall yield stress.

When using the vane tool or a cup and bob system for measuring yield stress it is important that the measurement is made at the bob wall or vane edge as opposed to the midpoint position, which is the DIN standard. This is because the stress decreases with radial distance from the bob surface and hence yielding will initially occur at the bob surface^{20, 23}. When using the vane tool the rotating vane will circumscribe a path in the sample and thus can be considered to behave like a cylindrical bob that is made out of sample, as shown in Figure 9. As sample is in contact with sample then minimum slip is encountered at the periphery of the vane, however, if the surface of the cup is not profiled then there is the possibility that the sample may slip or yield at the outer wall surface before the sample yields. Since stress decreases with inverse radius from the bob/vane surface, then using a larger measuring gap can minimize such effects. A serrated cup or basket may also be employed^{22, 23}.

CONCLUDING REMARKS

Yield stress is a critical parameter for characterizing a wide variety of complex fluids, and is a key factor for many real-life processes and applications involving such materials. In order to obtain relevant, robust and reproducible yield stress data for a particular material, it is essential to make an assessment of both the test type, and the measurement protocol used to perform the

test. It is this background understanding and consistency of approach that will make a difference to obtaining reliable yield stress measurements.

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