A Basic Introduction to Viscometers & Viscometry

Viscometer Types:

A viscometer (also called viscosimeter) is an instrument used to measure the viscosity and flow parameters of a fluid.

Glass viscometers

The classical method of measurement due to Stokes, consisted of measuring the time for a fluid to flow through a capillary tube. Refined by Cannon, Ubbelohde and others, the glass tube viscometer is still the master method for the standard determination of the viscosity of water. The viscosity of water is 0.890 mPa·s at 25 degrees Celsius, and 1.002 mPa·s at 20 degrees Celsius.

Rotational viscometers

Rotational viscometers use the idea that the force required to turn an object in a fluid, can indicate the viscosity of that fluid. The viscometer determines the required force for rotating a disk or bob in a fluid at known speed. 'Cup and bob' viscometers work by defining the exact volume of sample which is to be sheared within a test cell, the torque required to achieve a certain rotational speed is measured. There are two classical geometries in "cup and bob" viscometers, known as either the "Couette" or "Searle" systems - distinguished by whether the cup or bob rotates. 'Cone and Plate' viscometers use a cone of very shallow angle in theoretical contact with a flat plate. With this system the shear rate beneath the plate is constant to a modest degree of precision, a graph of shear stress (torque) against shear rate (angular velocity) yields the viscosity.

Rotational viscometers fall into two main types:

1. Synchronous (Stepper) Motor / Spring
2. Servo Motor / Digital encoder

The first type uses a stepper motor to drive the main shaft. A spring & pivot assembly rotate on the shaft. The spindle or rotor hangs from this assembly. As the spindle rotates the spring is deflected by the viscosity of the sample under test.

The second type uses a precision servo motor to drive the shaft. The Spindle or rotor is attached directly to the shaft. High speed microprocessors measure the speed from a digital encoder and calculate the current required to drive the rotor at the test speed. The current required is proportional to the viscosity of the sample under test.
Basic rheological terms

The following sections explain the basic terms such as stress and strain that are used in rheology & viscometry

Shear Strain.

To define the term STRAIN we will consider a cube of material with its base fixed to a surface as shown below in figure-1.

![Figure-1](image1)

If we now apply a constant 'pushing' force, F, to the upper part of the cube, assuming the material behaves as an ideal solid, it will obey Hooke's law of elastic deformation and will deform to a new position as shown in figure-2.

This type of deformation (lower fixed upper moving) is defined as a SHEAR DEFORMATION.

The deformation δ u and h are used to define the SHEAR STRAIN as:

\[ \text{Shear Strain} = \frac{\delta u}{h} \]

The shear strain is simply a ratio of two lengths (displacement / gap) and so has no units. It is important since it enables us to quote pre-defined deformations without having to specify sizes of sample etc.

Shear Stress.

The SHEAR STRESS is defined as \( F/A \) (A is the area of the upper surface of the cube \( l \times w \)) Since the units of force are Newtons and the units of area are \( m^2 \) it follows that the units of Shear Stress are \( N/m^2 \) This is referred to as the PASCAL (i.e. \( 1 \text{ N/m}^2 = 1 \text{ Pascal} \)) and is denoted by the symbol \( \sigma \) (in older textbooks you may see it denoted as \( \tau \))

Shear Rate.

Consider the case of a cube of material that behaves as an ideal fluid. When we apply a shear stress (force) the material will continually deform at a constant rate as illustrated in figure-4.

![Figure-4](image2)

The rate of change of strain is referred to as the SHEAR STRAIN RATE often abbreviated to SHEAR RATE and is found by the rate of change of strain as a function of time i.e. the differential \( \frac{d\text{Shear Strain}}{d\text{TIME}} \)

Typical Shear rate's for some standard processes

<table>
<thead>
<tr>
<th>Process</th>
<th>Typical range (S-1)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Spraying</td>
<td>( 10^4 ) to ( 10^5 )</td>
</tr>
<tr>
<td>Rubbing</td>
<td>( 10^4 ) to ( 10^5 )</td>
</tr>
<tr>
<td>Curtain coating</td>
<td>( 10^2 ) to ( 10^3 )</td>
</tr>
<tr>
<td>Mixing</td>
<td>( 10^1 ) to ( 10^3 )</td>
</tr>
<tr>
<td>Stirring</td>
<td>( 10^1 ) to ( 10^3 )</td>
</tr>
<tr>
<td>Brushing</td>
<td>( 10^1 ) to ( 10^2 )</td>
</tr>
<tr>
<td>Chewing</td>
<td>( 10^1 ) to ( 10^2 )</td>
</tr>
<tr>
<td>Pumping</td>
<td>( 10^0 ) to ( 10^3 )</td>
</tr>
<tr>
<td>Extruding</td>
<td>( 10^0 ) to ( 10^2 )</td>
</tr>
<tr>
<td>Sagging</td>
<td>( 10^0 ) to ( 10^2 )</td>
</tr>
<tr>
<td>Levelling</td>
<td>( 10^{-1} ) to ( 10^{-2} )</td>
</tr>
<tr>
<td>Sedimentation</td>
<td>( 10^{-1} ) to ( 10^{-3} )</td>
</tr>
</tbody>
</table>

Viscosity

The Shear Rate obtained from an applied Shear Stress will be dependant upon the materials resistance to flow i.e. its VISCOSITY.

Since the flow resistance \( \equiv \text{force / displacement} \) it follows that;

\[ \text{VISCOSITY} = \frac{\text{SHEAR STRESS}}{\text{SHEAR RATE}} \]

The units of viscosity are \( \text{Nm}^2\text{s} \) Which are better known as Pascal Seconds (Pa.s).

If a material has a viscosity which is independent of shear stress then it is referred to as an ideal or NEWTONIAN fluid. The mechanical analogue of a Newtonian fluid is a viscous dashpot which moves at a constant rate when a load is applied as seen in figure-5.
Material Approximate Viscosity (Pa.S)

Air 10^-5
Acetone (C3H6O) 10^-4
Water (H2O) 10^-3
Olive Oil 10^-1
Glycerol (C3H8O3) 10^+0
Molten Polymers 10^+3
Bitumen 10^+8

Basic Viscometry Units:

Viscosity
1 Ns/m^2 = 1 Pa.s = 10 Poise

Viscosity in Poise / Density = Kinematic viscosity in Stokes

Shear Stress
1 Pa = 1 N/m^2 = 10 dyn cm^-2

Torque
1 mNm = 9.81 gcm = 10000 dyne cm

Selecting Measuring Systems

Measuring systems fall into three basic categories. These are:

(1) Cone and Plate
(2) Parallel Plates
(3) Cup and bob

Each type has its associated advantages and disadvantages which will be described in the following sections.

Cone and plate

This is in many instances the ideal measuring system. It is very easy to clean, requires relatively small sample volumes and with a little care can be used on materials having a viscosity down to about ten times that of water (10 mPa.S) or even lower.

Cone angles

Since strain and shear rate are calculated using the angular displacement and the gap it follows that the smaller the cone angle, the greater the error is likely to be in gap setting and hence your results. By using a relatively large angle (4° or 5°) it becomes easier to get reproducibility of gap setting. Unfortunately, the larger the cone angle the more the shear rate across the gap starts to vary.

In considering what cone angle to use it is worth looking at variations of shear against the gap compared to reproducibility of gap setting. The following table of expected errors comes from work by Adams and Lodge.

<table>
<thead>
<tr>
<th>CONE ANGLE VARIATION OF SHEAR TYPICAL ERROR IN (O) RATE ACROSS GAP % CALCULATIONS %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 0.03 0.02</td>
</tr>
<tr>
<td>2 0.21 0.08</td>
</tr>
<tr>
<td>3 0.28 0.18</td>
</tr>
<tr>
<td>4 0.49 0.32</td>
</tr>
<tr>
<td>5 0.77 0.50</td>
</tr>
<tr>
<td>7 1.5 0.98</td>
</tr>
<tr>
<td>10 3.1 2.0</td>
</tr>
</tbody>
</table>

This shows that for a 4° cone the shear rate will vary by less than 0.5% across the gap giving data with around 0.3% error. If a smaller cone angle is used, although the shear distribution error is small, the operator to operator gap settings could easily
introduce errors of over 5% even by experienced operators and so the larger angle gives a more acceptable error since it is a reproducible error.

**When NOT to use a cone and plate.**

Because of the importance of correct positioning (often referred to as ‘gap setting’) a cone and plate is not recommended when performing temperature sweeps or step changes unless your rheometer is fitted with an automatic system for thermal expansion compensation.

You should also avoid using a cone if the sample you are testing contains particulate material. If the mean particle diameter is not some five to ten times smaller than the gap, the particles can ‘jam’ at the cone apex resulting in noisy data.

Materials with a high concentration of solids are also prone to being expelled from the gap under high shear rates, another reason to avoid the use of the cone.

**Parallel plate**

The parallel plate (or plate-plate) system, like the cone and plate, is easy to clean and requires a small sample volume. It also has the advantage of being able to take pre-formed sample discs which can be especially useful when working with polymers. It is not as sensitive to gap setting, since it is used with a separation between the plates measured in mm. (See Figure-7)

Because of this it is ideally suited for testing samples through temperature gradients.

**Figure-7**

**Advantages and disadvantages of parallel plates**

The main disadvantage of parallel plates comes from the fact that the shear rate produced varies across the sample. In most cases you will find that your software actually takes an average value for the shear rate.

Note also that the wider the gap, the more chance there is of forming a temperature gradient across the sample and so it is important to surround the measuring system and sample with some form of thermal cover or oven.

Parallel plate systems are referred to by the diameter of the upper plate. For instance, a PP40 is a 40mm diameter plate. The lower plate is either larger than or the same size as the upper plate.

When it is important to test samples at a known shear rate for critical comparisons the use of Parallel plates is not recommended.

**Sample loading for cone and plate and parallel plate measuring systems.**

The sample should just fill the gap between the upper and lower elements. If the sample is likely to shrink during the test (due to solvent loss etc.) it is advisable to aim for a slight bulge as shown in Figure-8. If too much or too little sample is used, the torque produced will be incorrect leading to the data being higher or lower respectively.

**Figure-8**

**Using stiff materials**

When using stiff materials with parallel plates, the best results can often be obtained by pre-forming the sample into a disc of the same diameter of the upper plate.

The thickness should be very slightly thicker than the required value so that the plates may be brought down such that they slightly compress the material, thus ensuring a good contact.

**Preventing solvent loss / drying**

Some samples may be prone to skinning or drying. This will happen at the edge of the sample to its exposure to atmosphere. To overcome this fit a solvent trap to the measuring system.

Another technique is to apply a fine layer of low viscosity (approximately 10 times thinner than the sample) silicon oil around the measuring systems. This works well provided that the oil and sample are not miscible and also that relatively small rotational speeds are being used so as not to mix the oil into the sample.

**Cup and Bob (Couette)**

Cup and bob type measuring systems come in various forms such as coaxial cylinder, double gap, Mooney cell etc. as shown in figure-9.

**Figure-9**

For DIN standard coaxial cylinders they are referred to by the diameter of the inner bob. i.e. a C25 is a coaxial cup and bob having a 25mm diameter bob. The diameter of the cup is in...
The double gap measuring system has the largest surface area and is therefore ideal for low viscosity / low shear rate tests.

Preventing solvent loss / drying

Some test materials may be prone to 'skinning' with time due to sample evaporation etc. To overcome this fit a solvent trap onto the measuring system.

Another technique is to float a very low viscosity (10 to 100 times thinner viscosity) silicon oil on the top of the sample in the cup. This works well provided that the oil and sample are not miscible and also that relatively small rotational speeds are being used so as not to mix the oil into the sample.

Rules of thumb for shear rate / shear stress selection.

Decrease cone/plate diameter to increase available shear stress.

Decrease bob surface area to increase shear stress

Decrease cone angle (or gap in a parallel plate) to increase available shear rate

(Remember: smaller the angle the more difficult to set gap correctly)

Use large surface areas for low viscosity and small surface areas for high viscosities.

Summary of measuring system selection.

Thick materials can be tested with a cone and plate unless they contain particulate matter, in which case use a parallel plate (remember that the shear rate will then only be an averaged value).

If you are performing a temperature sweep, use a parallel plate in preference to a cone and plate due to variations in the gap with thermal expansion of the measuring system.

For low viscosity materials and mobile suspensions use a cup and bob type system. Maximum sensitivity is obtained with a double concentric cylinder (double gap).

For oscillatory measurements at high frequencies on low viscosity materials, the C25 cup and bob or a parallel plate with a small gap will produce the optimum test conditions.

For testing low viscosity materials when only small sample volumes are available, use a Mooney Cell (such as a 'small sample cell')

For all samples, if drying or skinning of the sample is likely to be a problem, use a solvent trap with the measuring system or alternatively use a low viscosity silicon oil as a barrier if it is not likely to alter the samples properties.

Types of Viscometry test, Flow Characterization

The next section covers the definitions and explanation of basic flow characterization techniques.

The viscometry test

There are generally two types of simple flow characterization tests for viscometry. These are Stepped shear rate or Constant shear rate.

The types available on your particular instrument will depend upon the configuration of your rheometer software.

Stepped shear. Individual shear values are selected. Each shear is applied for a user set time and the shear rate, shear stress and viscosity are recorded for each value.

The individual points are then either joined up 'dot to dot' fashion or using a rheological model to produce the flow curve and the viscosity curve.

This test method is the generally the preferred way of generating flow and viscosity curves.

Types of flow curve

The measured viscosity of a fluid can often be seen to behave in one of four ways when sheared, namely:

1. Viscosity remains constant no matter what the shear rate (Newtonian behavior)
2. Viscosity decreases as shear rate is increased (Shear thinning behavior)
3. Viscosity increases as shear rate is increased (Shear thickening behavior)

4. Viscosity appears to be infinite until a certain shear stress is achieved (Bingham plastic)

Over a sufficiently wide range of shears it is often found that the material has a more complex characteristic made up of several of the above flow patterns.

**Flow curves**

Since it is the relationship of shear stress to shear rate that are strictly related to flow we can directly show the flow characteristics of a material by plotting shear stress vs shear rate. A graph of this type is called a Flow Curve. Figure-10 show the flow curves and viscosity curves of the four basic flow patterns.

![Flow Curves](https://www.rheosys.com/intro.html)

The exact behavior of materials can often be described by some form of rheological model. Some of the more commonly used models are described in the following section.

**Models for fundamental flow behavior**

These models describe the simple flow behavior as shown in the previous graphs.

Most materials will start to deviate from these relationships over a sufficiently large shear range.

They are well suited to studying materials over a small shear range or where only a simple relationship is required.

**Newtonian**

This is the simplest type of flow where the materials viscosity is constant and independent of the shear rate. Newtonian liquids are so called because they follow the law of viscosity as defined by Sir Isaac Newton;

Shear Stress = Shear rate * viscosity

Water, oils and dilute polymer solutions are some examples of Newtonian materials.

**Power law - (or Ostwald model)**

Many non-Newtonian materials undergo a simple increase or decrease in viscosity as the shear rate is increased. If the viscosity decreases as the shear rate is increased the material is said to be "shear thinning" or "pseudo plastic". The opposite effect is known as shear thickening. Often this thickening is associated with a change in sample volume. This is called "dilatency".

The Power law is good for describing a materials flow under a small range of shear rates. Most materials will deviate from this simple relationship over a sufficiently wide shear rate range.

Shear stress = viscosity * shear rate ^ n

Where 'n' is often referred to the Power law index of the material.

If n is positive, material is shear thinning, if n is negative then material is shear thickening.

Polymer solutions and melts as well as some solvent based coatings show Power law behavior over limited shear rates.

**Bingham**

Some materials exhibit an 'infinite' viscosity until a sufficiently high stress as applied to initiate flow. Above this stress the material then shows simple Newtonian flow. The Bingham model covers these materials;

Shear Stress = Limiting shear Stress + viscosity*shear rate

The limiting stress value is often referred to as the Bingham "yield stress" or simply the "yield stress" of the material. It should be noted that there are many definitions of Yield stress. For further information on this topic see the section on Yield values later.

Many concentrated suspensions and colloidal systems show Bingham behavior.

**Herschel Bulkley**

This model incorporates the elements of the three previous models

Shear stress = limiting stress + viscosity * shear rate^n

Special Cases of the model:
A pure Newtonian material has limiting stress=0 and n=0
A power law fluid has limiting stress=0 and n=power law index
A Bingham fluid has limiting stress= 'Yield value' and n=0

This models many 'industrial' fluids and so is often used in specifying conditions in the design of process plants.

**Calculation of Shear Rate and Shear Stress form factors.**

Rheometers and viscometers work with torque and angular velocity. Since we normally work with shear stress and shear rates a method is needed to convert from 'instrument numbers' to 'rheology numbers'.

Each measuring system used in an instrument will have its associated 'form factors' to convert torque to shear stress and to convert angular velocity to shear rate.

We will call the shear stress form factor C1 and the shear rate factor C2

\[
\text{Shear Stress} = C_1 \times \text{Torque} \\
\text{Shear Rate} = C_2 \times \text{angular velocity} \\
\text{Viscosity} = \frac{\text{Shear Stress}}{\text{Shear Rate}}
\]

* For some measuring systems such as parallel plates, the gap between the measuring systems can be set by the user. In this case the equation used is :
\[
\text{Shear Rate} = \frac{C_2 \times \text{angular velocity}}{\text{gap}}.
\]

The following section shows how the form factors are calculated for each measuring system.

**Cone and plate**

\[
C_1 = \frac{1}{2/3 \pi r^3} \\
C_2 = \frac{1}{\theta}
\]

Where \( r \) = radius of cone \( \theta \) = cone angle in radians

**Parallel plates (with 1mm gap)**

\[
C_1 = \frac{1}{2/3 \pi r^3} \\
C_2 = \frac{3r}{4}
\]

Where \( r \) = radius of plate

NOTE: The shear stress varies across the radius for a parallel plate. The above formula refers to the 3/4 radius position if the test sample is Newtonian.

**Coaxial cylinders**

\[
C_1 = \frac{1}{2 \pi r \alpha^2 H} \\
C_2 = \frac{2 \alpha r^2}{\alpha^2 (r^2 - r_0^2)}
\]

Where \( r_0 = (r_i + r_o) / 2 \)
\( r_i \) = inner radius
\( r_o \) = outer radius
\( H \) = height of cylinder

NOTE: \( C_1 \) takes the shear stress as that occurring at an average radius position \( r_0 \)

If you have any questions, please feel free to contact us at info@rheosys.com