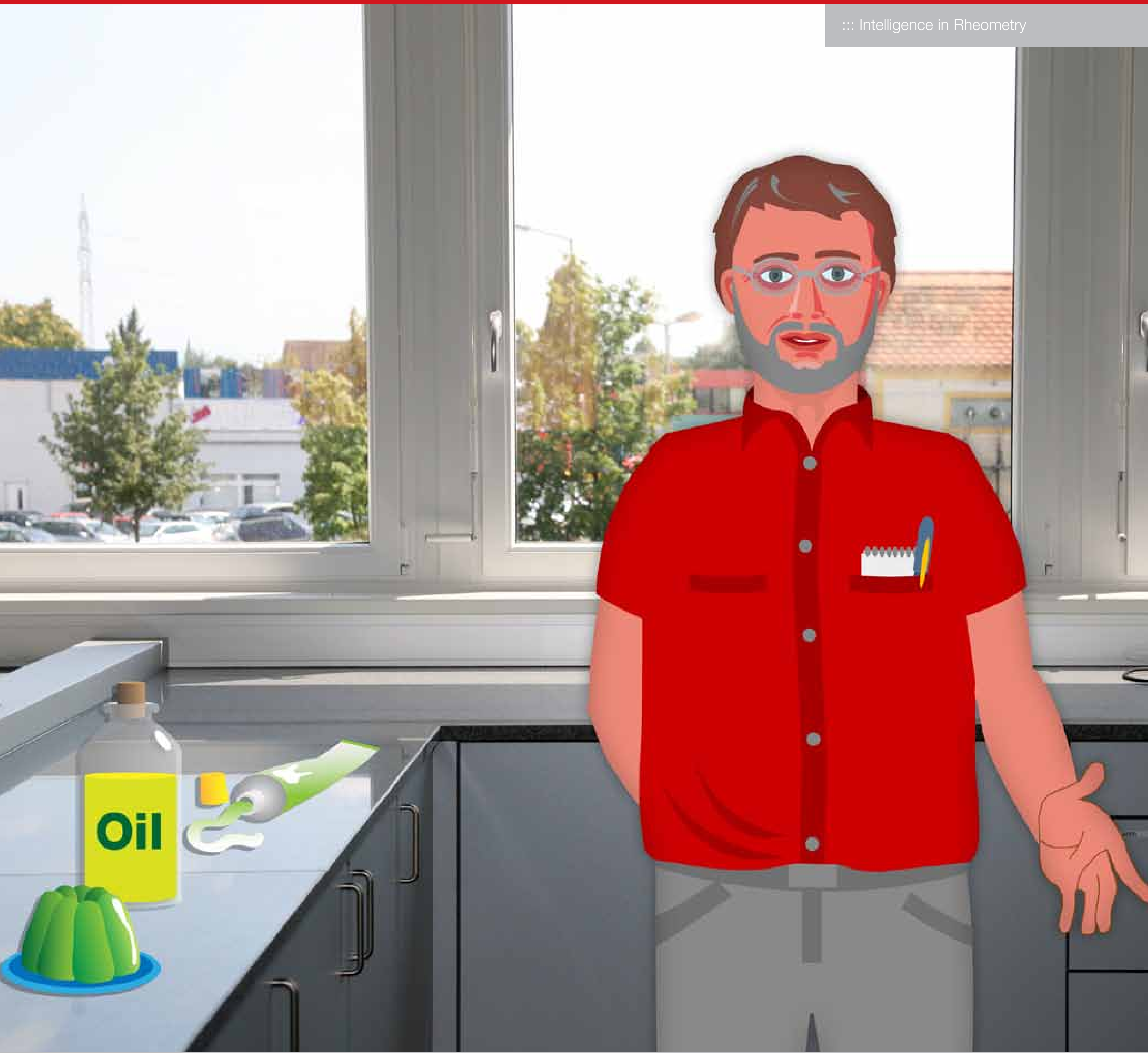
Anton Paar

::: Intelligence in Rheometry



Tips and Tricks from Joe Flow

Turn to rotational measurements!

With the right know-how you can quickly and easily characterize the viscous properties of your samples using rotational tests.

What is a rotational measurement and what are the most frequently used types of rotational measurements?

With rotational measurements the measuring bob turns in one direction while the lower plate or measuring cylinder does not move. This causes laminar flow in the shear gap of the sample.

There are different types of rotational tests. In these tests there are two different types of test settings which can be used:

- In a controlled shear rate test (CSR or CR test) the speed or shear rate is set and controlled and the torque or shear stress is measured.
- In a controlled shear stress test (CSS or CS test) the torque or shear stress is set and controlled and the speed or shear rate is measured.

The viscosity is calculated using the formula:

$$\textit{Viscosity}\ \eta = \frac{\textit{Shear stress}\ \tau}{\textit{Shear rate}\ \dot{\gamma}}$$

The different test types:

- **Single-point measurements:**
  In this test type the viscosity is measured at constant settings. This measurement is only suitable for Newtonian substances, whose viscosity remains constant independent of the set load. However, it is often used for quality control of non-Newtonian substances.
- **Time test:**
  This test is used to evaluate the time dependence of a sample under constant settings, e.g. during curing or gelification.
- **Flow and viscosity curves:**
  To produce flow and viscosity curves a shear rate range is set and the viscosity is measured as a function of the shear rate. With a flow curve the shear stress τ is usually plotted on the y-axis and the shear rate $\dot{\gamma}$ on the x-axis. With a viscosity curve, the viscosity η is usually plotted on the y-axis and the shear rate $\dot{\gamma}$ on the x-axis. Most samples show shear-thinning behavior. This means the viscosity decreases with increasing shear rate. Figure 1 gives an overview of the viscosity functions for substances without a yield point.

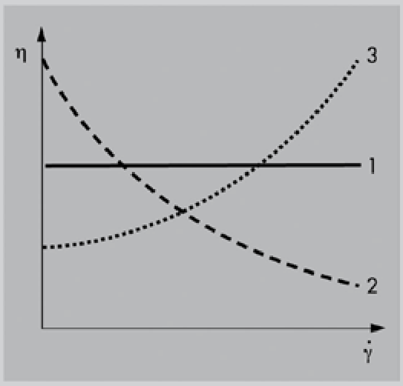


Fig. 1: Viscosity functions: (1) ideally viscous or Newtonian flow behavior, (2) shear-thinning flow behavior, (3) shear-thickening flow behavior

- **Step test:**
  This test is predominantly used to determine thixotropy, which is the structural decomposition and regeneration of a substance This is the topic of the next "Tips and Tricks from Joe Flow".
- **Temperature test:**
  Under constant settings the viscosity is determined as a function of the temperature.

Which settings should I set for my flow and viscosity curves?

Generally I recommend measuring under conditions which reflect reality as closely as possible. This realistically simulates technical processes.

There are many measuring procedures available in the RheoPlus software. You can change and expand these as required, e.g. combining intervals or adding rest or waiting periods.

To determine the flow behavior of a sample I recommend using a logarithmic setting for the shear rate. This means the measuring points are evenly distributed over the decades, which leads to more points being measured in the lower shear rate range. (When you look at the graph, it is like having a magnifying glass in your hand). For unknown samples, for example, a shear rate range of 0.1 s^{-1} to 100 s^{-1} has proved useful. If you select, for example, 22 measuring points, you will have 7 measuring points in every decade. This means 7 measuring points in the shear rate range between 0.1 s^{-1} and 1 s^{-1}, 7 between 1 s^{-1} and 10 s^{-1} and 7 between 10 s^{-1} and 100 s^{-1}. If you select a linear setting for the same shear rate range with the same number of measuring points, the second measuring point is already at approx. 5 s^{-1}. In contrast, you will obtain a high number of measuring points at higher shear rates. However, the curve at higher shear rates is often described adequately with less measuring points. Usually the behavior of the sample at low shear rates is interesting because this is when the viscosity changes the most, particularly with samples with a yield point.

Figures 2 and 3 show two viscosity curves (linear and logarithmic set shear rate, same number of measuring points) on a linear and logarithmic scale.

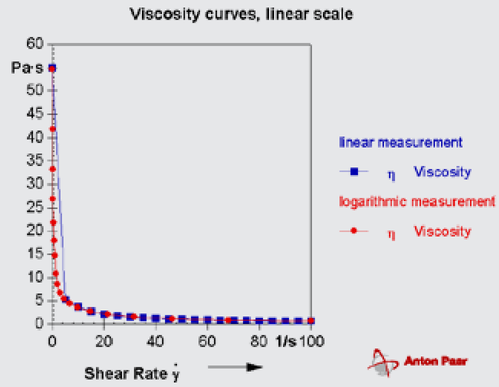


Fig. 2: Linear representation of two viscosity curves (linear and logarithmic set shear rate, same number of measuring points)

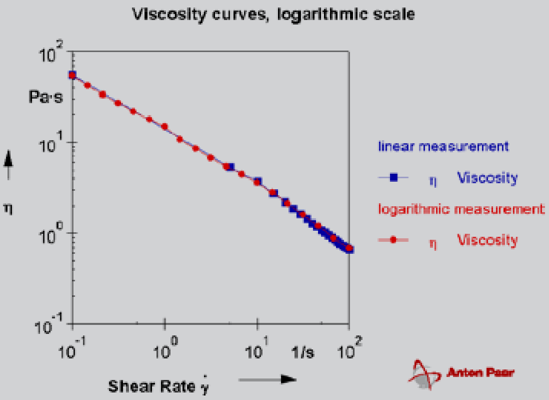


Fig. 3: Logarithmic representation of two viscosity curves (linear and logarithmic set shear rate, same number of measuring points)

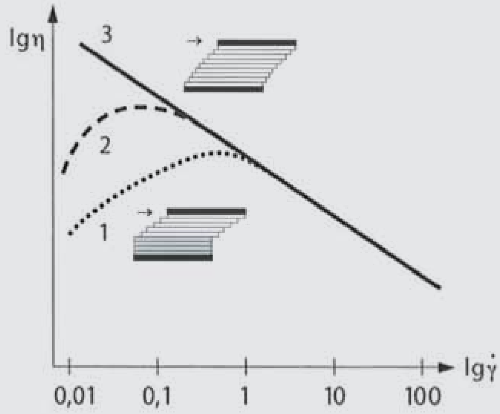


Fig. 5: Viscosity curve of a dispersion, occurrence of time-dependent effects (transient viscosity peak) at low shear rates and a measuring point duration which is too short (1), (2) and adequately long (3)

What do I need to consider for my measurements?

When shearing starts the sample begins to flow and a laminar flow forms (Fig. 4). This takes time (transient effects). The choice of the measuring point duration is therefore very important.
If the measuring point duration is too short the sample will not be completely sheared in the measuring gap (Fig. 4, middle diagram) and the rheometer will determine a viscosity which is too low and therefore incorrect.

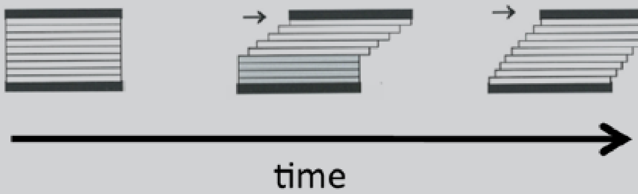


Fig. 4: Laminar flow as it forms during shearing

For shear rates under 1 s^{-1} the following rule of thumb has proved useful for the measuring time per measuring point: Measuring point duration = 1 / shear rate.
In the shear rate range from 0.1 s^{-1} to 100 s^{-1} a time of 10 s is required for the first measuring point at a shear rate of 0.1 s^{-1}. With increasing shear rate the time required for forming an even laminar flow becomes shorter and the measuring point duration can also be set to be shorter. When setting a logarithmic ramp for the shear rate it is therefore also useful to set an "inverse" logarithmic ramp for the measuring point duration. This ensures that the test does not run unnecessarily long. For shear rates larger than 1 s^{-1} I recommend a measuring point duration of 1 s.

Figure 5 shows the viscosity curves of a dispersion with a measuring point duration which is too short (Curve 1 and 2) and adequately long (Curve 3).

Measuring errors can have many causes and can usually be detected in the viscosity curve. They are often even more obvious in the flow curve. This was one of the topics in the previous "Tips and Tricks from Joe Flow".

Flow and viscosity curves with set shear stress

Flow and viscosity curves, as with all other rotational tests, can also be measured with controlled shear stress (CSS test). Figure 6 shows two flow curves with linear set CSR and CSS.

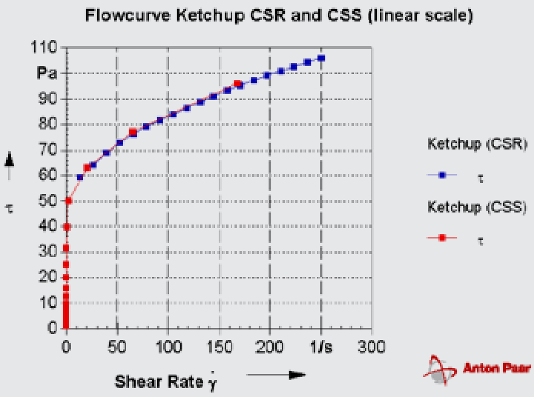


Fig. 6: Comparison of flow curves measured with set CSS and CSR

When the sample is flowing and the shear stress is set, very high shear rates may occur and the resulting centrifugal forces may lead to gap emptying. **For this reason, CSR tests are usually the preferred option today.** If you place a shear stress of 1 Pa on water (viscosity approx. 1 mPas), for example, and also on glycerine (viscosity approx. 1 Pas), the resulting shear rate with water is approx. 1000 s^{-1} and with glycerine approx. 1 s^{-1}.

CSS tests are sometimes still used to determine the yield point. Further information on the subject of yield point determination will be available in one of the upcoming "Tips and Tricks from Joe Flow".