UNIVERSITY OF SOUTH CAROLINA
ECHE 460 CHEMICAL ENGINEERING LABORATORY I

Viscometry and Rheology

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1. Introduction:

In this experiment you will measure viscosity and determine the rheological characteristics of some common fluids. Frequently the viscosity of a liquid mixture may not be available from the literature. The viscosity is a physical property that characterizes the resistance to motion of a simple fluid [3]. For example, we call motor oil to be "more viscous" than water because the oil would take more time to drain out of a container than an equal volume of water, that is to say that the oil offers more resistance to flow than does water.

Viscosity is one of the several properties that are studied in the field of *rheology* (defined by Eugene Bingham (1929) as "the study of the deformation and flow of matter" [6]). It is perhaps the most important rheological property in the design and selection of equipment for flow of fluids through pipes and ducts. In an another application, dilute solution viscosity measurements can be used in determination of molecular weight of linear-chain polymer molecules [1]. It is of interest to note here that Albert Einstein (1906, 1911) as part of his Ph.D. dissertation had formulated a method using viscosity measurement as a means to obtain the size of solute molecules dissolved in a solvent.

Viscosity has been measured using many different types of viscometers. A Brookfield viscometer will be used for measurements in this experiment.

2. Objective:

The overall objective of this experiment is to learn to measure the viscosity of fluids and determine the model that best fits the experimental data. Furthermore, you are expected to understand the limitations of each model. Additional technical objectives will become evident from the remainder of the handout.

3. Theory:

3.1 Definition of viscosity

Consider a fluid enclosed between two large parallel plates of area A, and separated by a distance H. By applying a tangential force over the top plate, it is made to move at a constant velocity, V, while the bottom plate is maintained stationary. Assuming that the top plate has been in motion for a long period and that the fluid does not slip at the plate surfaces, a steady state velocity profile, shown in Figure 1, is established in the liquid. We also assume that the flow is laminar. Under these conditions, the force per unit area (F/A) required to continuously move the top plate at a constant velocity is proportional to the velocity divided by the distance between the plates:

$$\frac{F}{A} = \mu\left(\frac{V}{H}\right) = \mu\left(\frac{V-0}{H-0}\right) = \mu \times velocity\_gradient \qquad (1)$$

The proportionality constant 'μ' is called the *viscosity* of the fluid. F/A is referred to as the *shear stress* (represented by the greek letter, τ) [N/m^2 or Pa], and has the units of pressure. The velocity gradient over the entire thickness of the fluid is equal to (V/H) and is, therefore, constant.

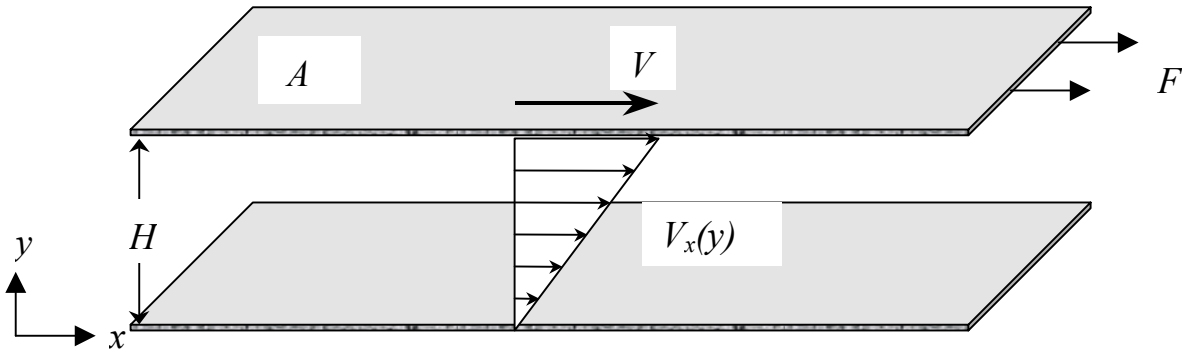

Figure 1. Steady simple shear flow of a fluid between two large parallel plates. The velocity field in this fluid is $v_x(y) = V.(y/H)$; $v_y = 0$; $v_z = 0$.

Now if we consider a small element of the fluid of thickness Δy and in which the velocity gradient is $_{\Delta}v_x$ along its thickness, by taking the limit $\Delta y \to 0$, Eq. 1 can be written for this fluid element as

$$\tau_{yx} = \lim_{\Delta y \to 0} \mu \frac{\Delta v_x}{\Delta y} = \mu \frac{dv_x}{dy} \tag{2}$$

where dv_x/dy is the velocity gradient (= V/B in Figure 1). The subscript "*yx*" has been added to τ to remind us that the shear stress is in the *x*-direction due to a velocity gradient in the *y*-direction. For the linear, constant velocity profile in Figure 1, we can rewrite the Eq. 2 as

$$\tau_{yx} = \mu \frac{dV_x}{dy} = \mu \dot{\gamma}_{yx} \tag{3a}$$

or equivalently as

$$\tau_{yx} = \mu \dot{\gamma} \tag{3b}$$

where $\dot{\gamma}_{yx}$ is the *rate-of-deformation* [s^{-1}], and $\dot{\gamma}$ is the *shear rate* [s^{-1}]. In this simple case the rate-of-deformation is equal to the shear rate.

The relationship between shear stress and the shear rate is linear in Eq. 3b, and this relationship is called the *Newton's law of viscosity*. The viscosity (μ, [Pa.s]) is equal to the ratio of the shear stress to the shear rate

$$\mu = \frac{\tau_{yx}}{\dot{\gamma}} \tag{4}$$

If the shear stress data are plotted against shear rate for a *Newtonian* fluid, the resulting curve is a straight line passing through the origin and with a constant slope μ. Thus, the viscosity of an incompressible Newtonian fluid is constant at a given temperature and pressure.

Several real materials are non-Newtonian fluids, *i.e.* the shear stress is not a linear function of shear rate. The slope of this curve is not constant and changes at every point along the curve, therefore, the slope can be said to be a function of shear rate (or shear stress) (it doesn't matter which way you want to define it). We have defined the viscosity as the ratio of shear stress to the shear rate; in the non-Newtonian case this ratio is referred to as the "apparent viscosity," and is represented by the symbol η [Pa.s].

$$\eta = \frac{\tau_{yx}}{\dot{\gamma}} \tag{5}$$

$$\tau_{yx} = \eta\,\dot{\gamma} = \eta\,\frac{dV_x}{dy} \tag{6}$$

Equation (6) is called the Generalized Newtonian model [3]. The apparent viscosity is not constant at a given temperature and pressure, but depends on the shear rate or, more generally, the shear history of the fluid [6].

3.2 Description of non-Newtonian behavior

Non-Newtonian fluids may be broadly classified into three types [6]: *time-independent, time-dependent,* and *viscoelastic*. Time-independent fluids are fluids for which shear stress is only a function of shear rate. Depending on the type of function these fluids can be subdivided into three types: *Bingham plastics*, *shear thinning*, and *shear thickening* fluids. Typical flow curves for these types of fluids are shown in Figure 2a. *Bingham plastics* do not flow until the applied stress exceeds a critical stress called the *yield stress* and when stresses exceeding the yield stress are applied the shear stress increases linearly with shear rate. Examples of Bingham plastics are drilling muds, oil paints, toothpaste, and sludges. Fluids in which the viscosity decreases at increasing shear rates, i.e., the ratio of shear stress to shear rate decreases at increasing shear rates are called *shear thinning* or *pseudoplastic* fluids. Many polymeric melts and solutions, dispersions and suspensions, *etc.* exhibit this behavior. *Shear thickening* or *Dilatant* fluids exhibit behavior exactly opposite to *shear thinning* fluids in that the viscosity increases as the shear rate is increased; however, this type of behavior is not common. In materials which exhibit *shear thickening* behavior, *shear thickening* is observed only in a limited-shear rate at high shear rates, and at other shear rates these materials usually exhibit *shear thinning* behavior. Figures 3-5 show experimental data for a few non-Newtonian fluids that have the behavior described above.

Time-dependent fluids, or fluids for which shear stress is not only function of shear rate but also the duration of shear. These fluids can be subdivided into two classes: *thixotropic* and *rheopectic* fluids. *Thixotropic* fluids are those for which the viscosity decreases with time when they are sheared at a constant shear rate. However, the trend is reverse in *rheopectic* fluids for which the viscosity increases with time when sheared at a constant shear rate. The behavior of these fluids is

illustrated in Figure 2b.

Viscoelastic fluids are fluids that have characteristics of both solids and liquids, and exhibit partial elastic recovery after deformation. Polymer melts and solutions, for example, exhibit this type of behavior. A more complete description of the non-Newtonian behavior in fluids can be obtained in the article by Patel [5]. It is **compulsory** for the student to read Patel's article. The student is also strongly recommended to read Chapter 1 of the textbook of Bird, Stewart and Lightfoot [3], and sections 1.1-1.3.2 in Chapter 1 of the textbook of Tanner [6].

The shear rate-dependent viscosity is not the only property that differentiates a Newtonian fluid from a non-Newtonian fluid. Interested readers are recommended to read Chapter 2 of the textbook by Bird, *et al* (1990) where the differences between Newtonian and non-Newtonian fluids are described with several photographs.

3.3. Models for viscosity

In this experiment, you will be dealing with Newtonian and non-Newtonian liquids. All fluids have time-independent viscosity. For some of the fluids you are asked to fit the experimental data obtained for these liquids to three models: Newtonian model, power law model, and Ellis model. The last two are non-Newtonian models applicable to *shear thinning* as well as to *shear thickening* fluids. These models are all well described in the attached article of Patel [5] and in standard references [2,3,6]. Note that the form of the Ellis model given in the above text books looks slightly different from the form you would be using in the data analysis (section 6), but essentially these two equations are the same, which is left for you to verify.

You will also measure the viscosity of dilute polymer solutions and will compute the molecular weight of the polymer from these measurements with the aid of the Mark-Houwink equation.

Newtonian model:

$$\tau = \mu \dot{\gamma} \tag{7}$$

If the steady state shear stress versus shear rate data fit a straight line passing through the origin, then the fluid is Newtonian.

Power-law model:

$$\eta = m\left|\dot{\gamma}\right|^{n-1} \tag{8}$$

The two parameters m and n are empirical parameters determined from experimental data. For shear thinning fluids $n < 1$ and for shear thickening fluids $n > 1$. To determine the two parameters, linearize the equation by taking the logarithm of both sides and perform linear regression of the data.

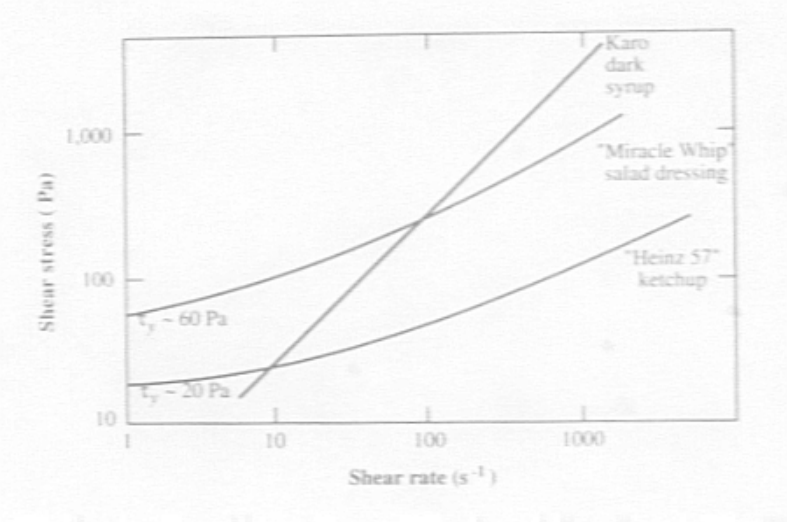

Figure 3. Flow data for several food products. (from Macosko, C. W., *Rheology: Principles, Measurements and Applications*, VCH Publishers, Inc., New York, 1994)

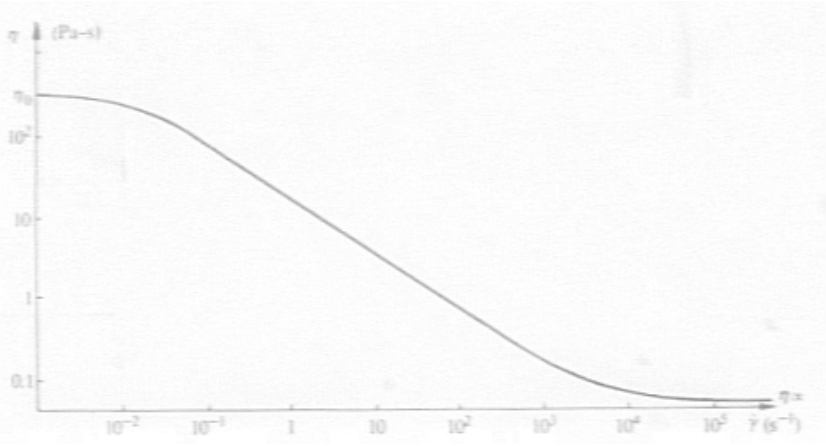

Figure 4. Shear-thinning in a polyacrylamide (Separan AP-30, DOW Chemical) in glycerol/ water solution [6].

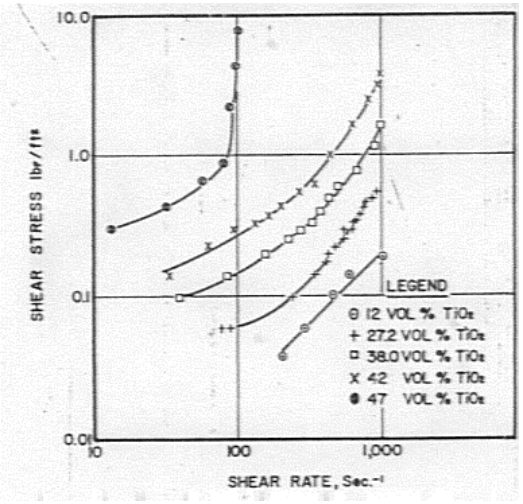

Figure 5. Shear thickening for aqueous suspensions of TiO_2 spheres, about 1μm diameter. (Metzner, A. B., and M. Whitlock, "Flow Behavior of Concentrated (Dilatant) Suspensions," Trans. Soc. Rheol. **2**, pp. 239-254 (1958)). Note that the shear stress (therefore, the viscosity) at a given shear rate increases with increase in volume % of TiO_2 spheres.

Ellis model:

$$\frac{\eta_0}{\eta} = 1 + \left(\frac{\tau}{\tau_{1/2}} \right)^{\alpha - 1} \tag{9}$$

The Ellis model is a three-parameter equation where η_0 is the zero-shear apparent viscosity, $\tau_{1/2}$ is the value of the shear stress when the viscosity is $\eta_0/2$, and α is the third parameter. Because the model has three parameters, one cannot simply do a linear regression to get the parameters. One way to approach the parameter estimation is to proceed in two steps. First, obtain the zero-shear apparent viscosity η_0 by fitting the shear stress versus shear rate, using only the lower shear rate data. At the lowest shear rates, the data will fit a straight line with a zero intercept. The slope of this line is η_0. Next, use all the data to obtain α and $\tau_{1/2}$. Take logarithms of equation 9 to linearize it, and plot $\ln(\eta_0/\eta - 1)$ vs $\ln(\tau)$. The plot should be linear. With the slope and intercept one can find the Ellis parameters.

3.4 Polymer molecular weights from dilute solution viscosity:

The rapid determination of polymer molecular weights is very important to industry. The Mark-Houwink relationship relates the *intrinsic viscosity* [η] (also called the *inherent viscosity*) to the polymer molecular weight M as follows:

$$[\eta] = K' M^{a}$$

The intrinsic viscosity is obtained from several measurements of polymer solution viscosity at increasingly dilute concentrations, as suggested by the following definition:

$$[\eta] = \lim_{c \to o} \frac{\eta - \eta_s}{c \eta_s}$$

where c is the concentration of the polymer in the solvent solution (g/dl) and η_s is the viscosity of the pure solvent. The Mark-Houwink equation has two parameters, K' and a, so that one must have data from polymers of two different molecular weights in order to obtain the parameters. Once the two parameters are known, one can obtain dilute solution viscosities of an unknown polymer and determine the molecular weight.

3.5. Measurement of viscosity:

A viscometer is defined as "an instrument for the measurement of viscosity" [8]. Two of the most commonly used instruments are the capillary viscometer and the rotary viscometer. A rotary viscometer can be used with one of the several geometries such as the cone-and-plate geometry, the

parallel-plate (circular discs) geometry, and the Couette (concentric-cylinders) geometry.

In the rotary-type of viscometers, the test fluid is confined in a thin film between two pieces of the instrument. The two pieces are precision-machined in a known geometry that can be modeled mathematically. One piece is rotated by a motor at a prescribed rotational speed (leading to a known shear rate), while the other piece is held stationary. One of the pieces is suspended to a torsion-bar, and the torque acting on this piece twists the torsion-bar (a spring, in the case of Brookfield viscometer) [8]. The Brookfield viscometer is a rotary-type instrument in which the rotating piece is directly attached to a copper-beryllium alloy spring. The most commonly used geometries are: cone-and-plate (see Figure 6), parallel plate (circular discs), and Couette (concentric cylinders).

The cone-and-plate apparatus is probably the most popular geometry for measuring the properties of liquids. The advantage with this geometry is that, for a given angular velocity *W*, the shear rate is constant throughout the liquid in the gap. This is true if the cone-angle, θ_0, is very small. Since the shear stress is a function of shear rate, a constant shear rate in the gap implies that the shear stress is also constant throughout the gap. Assuming that there is no slip at the walls, the shear rate and shear stress are related by:

$$\dot{\gamma} = \frac{W}{\theta_0} \tag{10}$$

Equation (10) does not contain a radial dimension, which implies that the shear rate is independent of the radius; hence, we say $\dot{\gamma}$ is constant everywhere in the gap. However, the shear rate is clearly a function of the angle θ_0, and the validity of the constant shear rate in the gap hinges on the assumption that θ_0 is very small. When $\theta_0 < 4.0°$, the error in assuming constant shear rate is very small. Most commercial cones available in markets have cone angles ranging from 0.5° to 4.0°.

Viscosity is the ratio of shear stress to shear rate, therefore to calculate the viscosity, information is needed on both the shear stress and the shear rate. The shear rate is computed from viscometer data using Eq. 10 above. The shear stress is determined by measuring the torque that is applied to the cone in order to sustain its rotation at a prescribed rotational speed. Again, the viscometer provides the torque data. The mathematical relationship between the applied torque and the shear stress is worked out below.

Consider a thin strip on the cone-surface of width *dr* and located at a distance of *r* from the apex of the cone. The differential torque, *dT*, acting on this strip is given by,

$$dT = |r||\mathrm{dF}|\sin\vartheta \tag{11}$$

where *dF* is the differential force acting on this strip, and ϑ is the angle between *r* and *dF* vectors (equal to 90° here). The differential force acting on this thin strip is a product of shear stress ($\tau_{\theta\phi}$) and the differential area of the strip. Therefore, Eq. 11 becomes

$$dT = r \times [\tau_{\theta\phi} \times (2\pi r dr)] \tag{12}$$

where $\tau_{\theta\phi}$ is the shear stress acting on this strip and ($2\pi r\, dr$) is the area of the strip (see Note 2). To determine the torque over the entire cone-surface, T, Eq. 12 is integrated with respect to r as follows:

$$T = \int_0^R \tau_{\theta\phi}\, 2\pi\, r^2\, dr \tag{13}$$

Because $\tau_{\theta\phi}$ is constant through out the gap and therefore is independent of r, it can be taken outside the integral (the subscripts on $\tau_{\theta\phi}$ are dropped because the shear stress is independent of position). Equation (13) now becomes:

$$T = \frac{2}{3}\pi R^3 \tau \tag{14}$$

Eq. 14 gives the torque (T) required to rotate the cone at a given angular velocity. For a more complete derivation (and a better analysis) of the above equation refer to example 3.5-3 in chapter 3 of reference 3, or example 1.3-4 in chapter 1 of reference 2. Rewriting the above equation with shear stress on the left hand side

$$\tau = \frac{3T}{2\pi R^3} \tag{15}$$

The viscosity can now be calculated using Eq. 16 as shown below:

$$\eta = \frac{\tau}{\dot{\gamma}} = \frac{3T}{2\pi R^3 \dot{\gamma}} \tag{16}$$

Note 1: The assumption that there is "no-slip" at the test surfaces is true in many cases, but it is not true for fluids whose flow is characterized by slip at the surfaces. Examples of fluids that exhibit wall-slip are mayonnaise, lubricants, concentrated suspensions and emulsions, and foams. Obtaining accurate rheological data for such systems is very complicated. Though you will not be dealing with fluids that may exhibit wall-slip in this experiment, you are advised to keep an open eye for such pitfalls in practical situations.

Note 2: This width of the (annulus) strip is so small ($r \gg dr$) such that it can be approximated to a rectangle of length equal to circumference of the strip ($2\pi r$) and breadth equal to the width of the strip (dr). Therefore, the area of the strip is given by the product of length ($2\pi r$) and breadth (dr).

Note 3: The cones available for testing, sometimes called *truncated* cones, are usually chopped (truncated) at the tip to avoid friction between its apex and the plate. It is therefore **important** to separate the cone- and plate by a distance equal to the height of the chopped part (~ 50 μm) if a cone-and-plate flow has to be achieved. How would it affect your viscosity measurements if the tip of the cone is not removed and the tip just touches the bottom plate?

4. Experiment:

Note: Please read the attached Operating Manual for the Brookfield viscometer before you start to perform the experiments below. Please work closely with your TA, and BE CAREFUL not to drop or damage the cone of the viscometer in any way! If the cone is scratched or dented, it will give unreliable viscosity readings.

a) Required Equipment and Supplies

Brookfield Digital Viscometer Model DV-II

CP-40 Cone for the Brookfield Viscometer

Temperature bath

Thermometer

Canon S 20 viscosity standard liquid

Glycerol

Deionized water

Carboxymethyl cellulose (CMC) powder

Polyacrylamide powder

Toluene solvent

Poly(methyl methacrylate) polymer standards of known and unknown molecular weights

b) Procedure

Part 1. Instrument/procedure check

The purpose of Part 1 is to check if the instrument is in proper working condition and to determine if your experimental technique is adequate. This is done by measuring the known viscosity of a standard liquid and by verifying that the measured viscosity agrees with the known value.

1. Set the temperature at 25°C in the controller on the temperature bath.
2. Using a pipette, place 0.5 ml of Canon S 20 viscosity standard liquid into the sample cup of the Brookfield viscometer (Step 8 on p. 4 of the Operations manual). The sample cup must be clean and dry prior to insertion of the sample. By gently shaking the cup, allow the sample to spread over the entire surface of the cup. Clip the sample cup to the adjusting ring, and allow the sample to stand for some time to reach the desired temperature (25°C).
3. At all rotation speeds possible with the instrument, measure the viscosity of the viscosity standard liquid. The value for viscosity is displayed in the digital dial. Record the percent torque (%), viscosity (CPS), and shear stress (SS) values displayed in the digital dial for the viscosity standard liquid.

The measured viscosity value at all rotation speeds should be the same (except when the "Low" indicator glows), and should be equal to the viscosity value specified on the bottle containing the viscosity standard. If not, move the adjusting ring by a very small distance in either direction, and repeat the measurements; if this doesn't work, then remove the cup and cone, clean and repeat the

procedure until this is achieved.

Part 2. Apparent viscosity as a function of shear rate for non-Newtonian fluids

In this part of the experiment you have to measure the viscosity as a function of shear rate for two liquids, and determine the type of their behavior. Furthermore, the data collected here will be used in Data Analysis to fit to three different models, and choose the model that best fits the data.

1. Prepare a 50/50 weight percent glycerol-water solution. Transfer a part of this well-mixed solution into another clean beaker, and to this add a measured weight of CMC powder (or polyacrylamide powder). The weight percent of the CMC in the glycerol-water mixture will be assigned to you later; typically it would be in the range of 0.5 to 2.5 weight percent%.

2. Following the above procedure, record the percent torque (%) and viscosity (CPS) values displayed in the digital dial at all rotation speeds for the 50/50 weight percent glycerol-water mixture. These measurements must all be conducted at 25 ^{o}C.

3. Similarly, record the percent torque and viscosity at 25°C for the solution of CMC (or polyacrylamide) in 50/50 weight percent glycerol-water mixture.

Part 3. Temperature dependence of apparent viscosity for a given fluid

1. At all rotation speeds, record the torque and viscosity values for the 50/50 weight percent glycerol-water mixture used in Part 2. Obtain data at three temperatures: 20 ^{o}C, 25 ^{o}C, and 30 ^{o}C.

Part 4. Molecular weight determinations using dilute solution viscometry

1. Obtain PMMA standards with two different molecular weights. Prepare three or four dilute solutions of the two polymers in the solvent toluene. The concentrations should be in the range of 1.2 g/dl and below.
2. Measure the viscosity of the standard solutions using the procedures outlined above.
3. You will be given a PMMA polymer of unknown molecular weight. Dissolve it in toluene and make the measurements necessary to determine its molecular weight.

Some precautions you need to take when performing the experiment:

- Repeat the mechanical procedure (steps 1-7 under “Operation” in the attached Operating Manual) each time the cone spindle is detached from the viscometer and then is reattached.
- Please switch "off" the motor whenever you are removing or replacing the sample cup.
- Make sure you add correct amount of liquid in the sample cup.

- Clean the cone and the cup with soapy water and rinse with deionized water after each experiment.

5. Data analysis:

1. Calculate the shear rate corresponding to each rotational speed. Then, using the percent torque obtained at each rotation speed, calculate the shear stress (τ) and the viscosity as a function of shear rate. Compare the calculated viscosity values with those read directly off the digital display of the Brookfield instrument. Remember that the calculated values and the corresponding values read from the digital display should match, otherwise check your calculations for possible errors.

2. Fit the shear stress-shear rate data (alternatively, you could use viscosity-shear rate data) taken at 25°C for the (a) 50/50 weight% glycerol-water mixture, and (b) CMC (or polyacrylamide) solution in 50/50 wt% glycerol-water mixture to the Newtonian, power law, and Ellis models given below. Select the model that best fits the data and briefly explain your choice. Also, explain clearly why the other models do not fit the data well.

3. Model the 50/50 wt% glycerol-water mixture date taken at different temperatures with the Arrhenius-type temperature-dependent model.

$$\mu = A\, e^{B/RT}$$

4. Estimate the parameters A and B. The above viscosity-temperature relationship is called Andrade equation [4], or Andrade-Eyring equation.

5. Use the dilute-solution viscosity data from the two solutions of known PMMA molecular weight to calculate the Mark-Houwink constants. Then use the M-H equation to estimate the molecular weight of the unknown PMMA sample.

Information you will need for data analysis:

Full-scale torque of the Brookfield instrument = 673.7 dynes-cm

Radius of the CP-40 cone = 2.4 cm

Angle of the CP-40 cone = 0.8° (convert into radians for calculations)

6. Reading material (compulsory)

Instruction manual for the operation of the Brookfield viscometer. This manual is extracted from the operational manual supplied by the manufacturer of this instrument.

Patel, R. D., "Non-Newtonian fluids,", pp 139-148, *Handbook of Fluids in Motion*, Eds. N. P.Cheremisinoff and R. Gupta, Butterworth Publishers, Stoneham, MA 02180 (1983).

Fox, T. G., S. Gratch, and S. Loshaek, "Viscosity Relationships for Polymers in Bulk and in Concentrated Solution," p 446-448, *Rheology: Theory and Applications*, Edited by F. R. Eirich, Academic Press Inc., Publishers, New York (1956).

7. Additional References:

(*Available in Thomas Cooper library)

1.* Billmeyer, F. W., Jr., *Textbook of Polymer Science*, 3rd ed., John-Wiley & Sons, New York, (1984).

2.* Bird, R.B., R.C. Armstrong and O. Hassager, *Dynamics of Polymeric Liquids, Vol. 1: Fluid Mechanics*, 2nd ed., John Wiley, New York (1990). (Note: 1st edition only is available)

3.* Bird, R.B., W.E. Stewart and E.N. Lightfoot, *Transport Phenomena*, John Wiley, New York (1960).

4.* Fox, T. G., S. Gratch, and S. Loshaek, "Viscosity Relationships for Polymers in Bulk and in Concentrated Solution,", p 446-448, *Rheology: Theory and Applications*, Edited by F. R. Eirich, Academic Press Inc., Publishers, New York (1956).

5.* Patel, R. D., "Non-Newtonian fluids,", pp 139-148, *Handbook of Fluids in Motion*, Eds. N. P. Cheremisinoff and R. Gupta, Butterworth Publishers, Stoneham, MA 02180 (1983).

6.* Tanner, R.I., *Engineering Rheology* , Oxford University Press (1988).

7.* Van Wazer, J. R., J. W. Lyons, K. Y. Kim, and R. E. Colwell, *Viscosity and Flow Measurement. A Laboratory Handbook of Rheology*, Interscience Publishers, New York (1963).

8. Walters, K., *Rheometry*, Chapman and Hall, London (1975).

Brookfield Viscometer: Operating Manual

1. Principle of Operation:

All Brookfield Digital Viscometers, including the Wells-Brookfield Cone/Plate Model DV-II, rotate a sensing element in a fluid and measure the torque necessary to overcome the viscous resistance to the induced movement. This is accomplished by driving the immersed element, which is called a spindle, through a beryllium-copper spring. The degree to which the spring is wound, detected by a rotational transducer, is proportional to the viscosity of the fluid.

Continuous readouts of percent full scale, viscosity and shear stress are provided by means of the integral three-digit LED display. The 0-10 mv, or the 0-1v analog output signal can be fed into a variety of indicating or recording devices, and the RS232 output can be connected to any suitable interface.

The Viscometer is able to measure over a number of ranges since, for a given spring deflection, the actual viscosity is inversely proportional to the spindle speed and shear stress is related to the spindle's size and shape. For a material of given viscosity, the drag will be greater as the spindle size and/or rotational speed increase. The minimum viscosity range is obtained by using the largest spindle at the highest speed; the maximum range by using the smallest spindle at the slowest speed.

Measurements made using the same spindle at different speeds are used to detect and evaluate the rheological properties of the test material.

2. Cone/Plate Theory

If the axis of a nearly flat conical surface is perpendicular to a flat plate with the cone's apex lying in the plane of the plate, and if either the cone or the plate is rotated with respect to the other about the axis, fluid in the space between the two will be subjected to uniform shear rate.

This, except for small edge effects, follows from the fact that the rate of movement of any point on either surface is proportional to its distance from the axis and that the separation of the surfaces at that point is equivalently proportional to the same radius. The ratio of the rate of movement of the surface (at any point) to the distance of separation is fixed for any speed of rotation, and constant over the entire surface. Since rate of shear is by definition this ratio, it is therefore constant.

By using small angles between cone and plate (less than 4°), substantial rates of shear and hence, shearing stresses, can be achieved with comparatively low rotational speeds, low viscosities and small samples.

3. Introduction

All Digital Viscometers are powered by a precision synchronous motor. Exact speeds of rotation are assured as the motor will turn erratically and spasmodically if synchronism cannot be maintained.

Speed changes are affected by a transmission having eight speeds. The round speed control knob rotates both clockwise and counter-clockwise. Maximum speed (rpm) will be set at full clockwise rotation and minimum speed at full counter-clockwise rotation. The speed setting is indicated by the number of the knob located opposite the button on the Viscometer housing. Although not absolutely necessary, it is advisable to change speeds while the motor is running.

Initialization

1. Turn power switch "on" (up), energizing Viscometer display. The power switch is on the left side of the front panel.

   The power switch should kept "on" at all times during the experiment irrespective of whether the motor is "on" or "off". If any time during the experiment, the power switch is turned "off", then remove the cup and detach the cone, and start all over again from step 3 onwards.

2. Check bubble level to be sure the Viscometer is level.

3. Make sure that the **cone** is not attached to the shaft. To detach the cone from the shaft, using the wrench supplied to you hold the lower shaft and lift it slightly, and then carefully unscrew the cone by rotating it in counter-clockwise direction.

4. Turn motor switch "on" (up) and set speed selector knob to 12 rpm. The motor switch is on the right side of the front panel.

5. Press AUTO ZERO and the Viscometer will zero position the electronics and pointer shaft displacement.

6. Turn motor switch "off", placing Viscometer in standby mode.

Operation

Cone/Plate Viscometers

1. Turn "on" temperature bath and allow sufficient time for sample cup to reach the desired temperature. Adjustments should always be performed at the operating temperature.

2. Swing sample cup clip to one side and remove sample cup. Using wrench supplied, hold Viscometer lower shaft and screw on cone spindle (in clockwise direction), lifting lower shaft slightly at the same time (note left-hand thread). Avoid putting side thrust on the shaft. Excess thrust on the shaft may cause a permanent damage to the Cu-Be spring.

3. Turn "on" the motor switch and set the speed selector knob to 60 rpm. Look for the smooth rotation of the cone. If the motion seems jerky or eccentric, turn "off" the motor, unscrew the cone and screw it back on the shaft. The mating surfaces of the spindle and lower shaft must be clean to prevent eccentric rotation of the spindle.

Turn "off" the motor if the cone rotates smoothly, and proceed to the next step.

4. Place sample cup against adjusting ring, being sure to position the notch on the side of the cup around the sample cup clip. Swing clip under cup to secure it in place.

   Avoid hitting the spindle when installing the sample cup. If the display doesn't return to zero after installing the sample cup, unscrew the adjusting ring (turn it to the left) until the display reading returns to zero.

5. Run the Viscometer at 12 rpm by setting the speed select knob and turning the motor switch "on".

   If the display reading regularly jumps to 0.3 or higher, or will not settle to zero (indicating that the pins in the spindle and the sample cup are contacting), screw the adjustment ring to the left until the reading stabilizes at or near zero.

   If the display reading remains at or near zero, continue to the next step.

6. Turn the adjusting ring to the right in small increments (one or two minor divisions on the ring) while watching the digital display. Turn the adjusting ring until fluctuation of the display reading indicates that the pins have made contact.
   Once contact has been made, back off the adjusting ring (turn it to the left) in small increments until stabilization of the display reading indicates that the pins are not contacting.

   Turn the adjusting ring to the right in very small increments (about 1/64") until the display reading fluctuates regularly by a small amount. This determines the point at which the pins are just making contact.

7. Make a pencil mark on the adjusting ring directly under the index mark on the pivot housing. Turn the adjusting ring to the left exactly the width of one minor division. This will separate the pins by exactly .0005".

   The viscometer is now mechanically set and ready for sample insertion.

   It is recommended that this mechanical procedure be performed every time the spindle is removed from the Viscometer and replaced. The Viscometer's calibration can be checked by the use of Brookfield Viscosity Standards (under controlled temperature conditions only) or any other calibrating fluids available in market.

8. Remove the sample cup. Place sample fluid in cup according to the table below, **being sure that the sample is bubble-free and spread evenly over the surface of the cup**. Sample volume must be sufficient to wet the entire face of the spindle and approximately 1.0 mm up the spindle's outside edge. **Make sure that you add correct amount of liquid.**

Spindle	Angle (degrees)	Sample Volume (ml)
CP-40	**0.8**	**0.5**
CP-41	3.0	2.0
CP-42	1.565	1.0
CP-51	1.565	0.5
CP-52	3.0	0.5

Replace the sample cup, being careful not to hit the spindle.

9. Allow sufficient time for the sample fluid to reach the desired temperature.

10. Press the SPDL key and enter the spindle number {**enter 40**} (refer to Appendix 1). After the two digit number is entered, press either the %, CPS or SS key.

11. To make a viscosity measurement, turn the motor switch "on", which energizes the Viscometer drive motor. Allow time for the display reading to stabilize. The time required for stabilization will depend on the speed at which the Viscometer is running and the characteristics of the sample fluid. The display reading stabilizes quicker at high speeds than at low speeds, therefore at low speeds it is advisable to take reading after allowing the viscometer to run for sufficiently longer times.

    The digital display on this Viscometer reads from 00.0-99.9 in the % mode. Overrange is indicated by **"EEE."** Underrange is "- - -." Floating point display is used for the viscosity **(CPS)** and shear stress **(SS)** modes. You can change modes at any time without affecting the viscosity measurement.

Low Reading Indicator

If the Viscometer reading is less than 10% of the full scale range, the low LED indicator will come on. The purpose of this indicator is to alert the operator that the measurement is on the low end of the full scale range. This is especially important when using the CPS and SS modes. The Viscometer will calculate viscosity and shear stress at any upscale reading above zero, and it is recommended to take readings above 10%.

12. Turn the Viscometer motor switch to "off" when changing or cleaning a spindle, changing samples, etc. This is a standby mode in which the electronic circuits of the Viscometer remain energized. It is advisable to leave the power switch "on" between tests to minimize drifting of the Viscometer reading.

    It is recommended, when operating the Viscometer for a lengthy period, that zero be checked occasionally as described previously. Remove spindle from the Viscometer before performing this procedure.

4. A Calibration Check

First verify that the Viscometer is running properly. People are often concerned about the accuracy of their Viscometer. Here are some tests of its mechanical performance.

(A) Variations in power frequency will cause the spindle to rotate at an incorrect speed. If you are in an area where electric clocks are used, this factor may be immediately eliminated. Voltage variations have no effect as long as the deviation is not greater than ± 10% of the nameplate voltage and the frequency remains constant.

Other readily apparent symptoms of improper power supply are: failure of the motor to start, jerky spindle rotation or inconsistent digital display readings.

(B) Damage to the pivot point or jewel bearing will adversely affect the accuracy and repeatability of the Viscometer. The following Oscillation Test will allow you to evaluate the condition of these components:

1. The Viscometer should be mounted and leveled, with no spindle installed and the motor switch in the "off" position.

2. Put the display into the % mode.

3. Turn the spindle coupling by hand to deflect the digital display upscale from its zero position to a reading of 5 to 10 and let it swing back under its own power.

4. If the coupling swings freely and smoothly, and the display returns to zero each time this test is repeated, the pivot point and jewel bearing are in good condition. If it crawls back sluggishly and does not come to rest on zero, the performance of the Viscometer will not be up to specification and it should be serviced.

(C) We have never found a spring made of beryllium copper which showed any change in its characteristics due to fatigue, even after hundreds of thousands of flexings. For this reason, a check of the calibrated spring is usually not necessary. The Auto Zero is provided to

compensate for any possible heat-induced drift in the electronic circuitry.

(D) The use of a calibrated viscosity standard is recommended as a final performance check. Test the viscosity standard as you would any sample fluid, carefully following any applicable instructions. The use of fluids other than viscosity standards is not recommended due to the probability of unpredictable rheological behavior.

(E) If the Viscometer passes all of the preceding tests, its performance should be satisfactory. Should the accuracy of operation of the instrument still be suspect, please refer to the troubleshooting suggestions.

5. Fault Diagnosis/Troubleshooting

The chart below lists some of the more common problems that you may encounter while using your Viscometer, along with the probable causes and suggested cures.

(A) Spindle does not rotate

1. Incorrect power supply
   · Check - must match Viscometer requirements
2. Viscometer not plugged in
   · Connect to appropriate power supply
3. Power switch in "off" position
   · Turn power switch on
4. Shift know set "between" speeds
   · Rotate knob to higher or lower speed setting

(B) Spindle rotates eccentrically

1. Spindle not screwed securely to coupling
   · Tighten
2. Dirt in spindle coupling
   · Clean
3. Bent spindle
   · Check other spindles - replace any that are bent
   · If all rotate eccentrically - see (B)4

Note: maximum permissible runout is 1/16 inch (1.6 mm) at end of spindle

(C) Display reads only "00.0"

1. No response to spindle deflection indicates 0-1v or 0-10mv output signal leads spindle
   · Check output connections
2. Hold key is on (light on)
   · Toggle hold off

(D) No display reading

1. Underrange "- - -" (in %, CPS or SS mode)
   · Change spindle and/or speed
   · Perform an Auto Zero
2. Spindle jammed
   · Consult factory or dealer

(E) Display reading over 100

1. Overrange "EEE" (in %, CPS or SS mode)
   · Change spindle and/or speed

(F) Viscometer will not return to zero

1. Pivot point or jewel bearing faulty
   · Perform calibration check
   · Return to factory or dealer for repair

(G) Display reading will not stabilize

1. Check for erratic spindle rotation - may be caused by incorrect power supply or mechanical fault
   · Return to factory or dealer for repair
2. Bent spindle or spindle coupling
   · Check
3. Temperature fluctuation in sample fluid
4. Characteristics of sample fluid

(H) Inaccurate readings

1. Incorrect spindle/speed selection
2. Incorrect Spindle (**SPDL**) entry
3. Non-standard test parameters
4. Temperature fluctuations
5. Incorrect equipment selection