

Viscosity Measurements of Barium Sulfate Mixtures for Use in Motility Studies of the Pharynx and Esophagus

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Abstract. Detailed viscosity measurements have been made of barium sulfate mixtures over a wide range of viscosities for use in radiography of the esophagus, stomach, and duodenum. A new methodology was developed for more accurate estimation of viscosity in non-Newtonian fluids in conventional cylinder-type viscometers. As base cases, the variation of viscosity with shear rate was measured for standard commercial mixes of e-z-hd (250% w/v) and a diluted mixture of liquid e-z-paque (40% w/v). These suspensions are strongly shear thinning at low shear rates. Above about $3s^{-1}$ the viscosity is nearly constant, but relatively low. To increase the viscosity of the barium sulfate mixture, Knott's strawberry syrup was mixed to different proportions with e-z-hd powder. In this way viscosity was systematically increased to values 130,000 times that of water. For these mixtures the variation of viscosity with temperature, and the change in mixture density with powder-syrup ratio are documented. From least-square fits through the data, simple mathematical formulas are derived for approximate calculation of viscosity as a function of mixture ratio and temperature. These empirical formulas should be useful in the design of "test kits" for systematic study for pharyngeal and esophageal

motility, and clinical analysis of motility disorders as they relate to bolus consistency.

Key Words: Viscosity — Bolus viscosity — Rheology — Shear rate — Viscometer — Viscometry — Deglutition — Deglutition disorders.

Abnormal motility during swallowing is commonly observed to be associated with the consistency of ingested food and drink. As a result, variation of bolus consistency is often used as a diagnostic tool in clinical trials [1-5]. More recently it has been recognized that systematic variation of both bolus volume and bolus viscosity can provide useful information for the study of the neuromuscular processes that control the swallowing process, and abnormalities that occur as these controls fail [6].

For use in studies of motility variation with bolus consistency, however, there exist very few quantitative data on systematic variations in the rheologic properties of contrast media. Specific discussions of barium sulfate as a contrast medium that mention viscosity as an important descriptive parameter [e.g., 7] fail to quantify viscosity for barium mixtures. Uses of adjectives such as "pasty" [8], "liquid," or "creamy" [e.g., 1] remain, of necessity, vague and unquantified. Indeed, one person's perception of pasty, liquid, creamy, and the like, may be very different from another person's perception. For a discussion of the relationship between oral

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sensation and bolus rheology, and some data on the viscosity of standard barium sulfate suspensions, saliva, and baby food, see [8].

Because this paper centers on viscosity as a primary rheologic variable, it is useful to remind the reader of the technical definition of viscosity as a quantitative measure of frictional resistance in a fluid to shear. Imagine placing the barium sulfate mixture between two large parallel plates with a narrow gap, as illustrated in Figure 1; the lower plate is held fixed and the upper plate pulled forward at constant fixed speed, U . The viscosity of the fluid is proportional to the force required to pull the upper plate to the same fixed speed. More precisely, the viscosity (μ) is given, in this example, by

$$\mu = \frac{F/A}{U/h} = F \times \left(\frac{h}{UA} \right) \quad (1)$$

where F is the force required to pull the plate, A is the surface area of the plate, U is the speed of the plate, and h is the gap spacing between the plates. Thus, for fixed plate geometry (A), gap spacing (h), and plate speed (U), if it takes twice as much force to pull the plate with one liquid as with another liquid, the viscosity of the first liquid is twice as high as the viscosity of the second liquid (where both plates are pulled at the same speed). To transport these two liquids through the esophagus where frictional forces tend to dominate, roughly twice as much power must be supplied by the musculature to transport the bolus that is twice as viscous (all other characteristics, such as bolus volume, geometry, wave speed, etc., being the same).

In the example of Figure 1, U/h is the “shear rate”: the rate at which the fluid is being sheared by the moving plate. If the fluid viscosity does not vary with shear rate it is called “Newtonian.” This is generally not the case with suspensions, since the attractive force between suspended particles weakens as the shear rate increases, leading to a reduction in effective viscosity with increasing shear rate. Such “non-Newtonian” fluids are called “shear thinning.” Our measurements show that commercial barium sulfate suspensions are shear-thinning at very low shear rates: below about $2\text{--}3\text{s}^{-1}$. At higher shear rates the fluid is Newtonian: its viscosity does not vary with shear rate. The effect of shear thinning is insignificant during pharyngeal transport since shear rates are very high there. However, shear thinning may play a role in esophageal barium bolus transport since bolus velocity is rather low and shear rates may vary between very small values near the center of the bolus to values on the order of 15s^{-1} at the esophageal wall. The importance of shear thinning

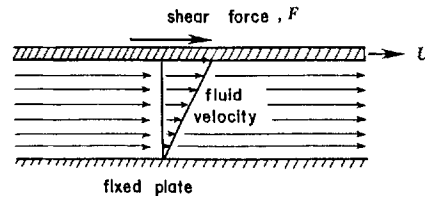


Fig. 1. Illustration of an experiment that quantifies “viscosity” as the frictional forces within the fluid resisting shear. The lower plate is held fixed and the fluid is inserted between the two plates. The shear force, F , is applied to the upper plate to move it at constant speed, U .

in esophageal bolus transport, however, has not been explored. In general, fluid viscosity is also dependent on temperature. Further discussion of bolus rheology in swallowing may be found in [9, 10].

In this paper we describe a series of systematic viscosity measurements of barium sulfate mixtures over a wide range of viscosities as a function of shear rate and temperature. The data are fitted with empirical formulas to allow individual users to create easily their own radiopaque “test kits” for radiologic motility studies and clinical trials.

A note on units: we use centipoise (cps) as the standard unit for viscosity. It is useful to keep in mind that the viscosity of water at room temperature is about 1 cps.

Measurement Protocols

Variation of Sample Mixture

The viscosities of the 10 sample mixtures listed in Table 1 were measured in detail. As baseline cases, the variations in viscosity with shear rate of standard mixes of *e-z-hd*[®] and a diluted mixture of Liquid *e-z-paque*[®] (*E-Z-EM*, Inc., Westbury, NY) were measured at room temperature. These samples could be qualitatively described as “liquidy,” and at high shear rates have viscosities around 200 cps. To increase the viscosity to thick, pasty consistencies in continuous increments, seven mixtures of *e-z-hd* powder and Knott’s strawberry syrup (Knott’s Berry Farm, Buena Park, CA) were formulated with different powder-to-syrup ratios. Two containers of *e-z-hd* powder were mixed with different volumes of syrup as shown in Table 1. The viscosity of Knott’s strawberry syrup alone was also measured. Knott’s strawberry syrup was first used by Dooley et al. [11] to increase the viscosity of barium samples, although systematic measurements of sample viscosity were not carried out. Knott’s syrup has the advantage of being easily obtainable and measurements

Table 1. Measurement samples

Name	e·z·hd powder weight (g)	Liquid volume
Standard e·z·hd mix	340	65 ml water (250% w/v)
Diluted liquid e·z·paque ^a	Premixed	40% w/v
Strawberry	0	Knott's strawberry syrup
Mixture 1	680 ^b	452 ml Knott's strawberry syrup
Mixture 2	680	400 ml Knott's strawberry syrup
Mixture 3	680	350 ml Knott's strawberry syrup
Mixture 4	680	300 ml Knott's strawberry syrup
Mixture 5	680	250 ml Knott's strawberry syrup
Mixture 6	680	200 ml Knott's strawberry syrup
Mixture 7	680	150 ml Knott's strawberry syrup

^a Diluted liquid e·z·paque was mixed with one part water to dilute the mixture to 40% w/v.

^b 680 g of e·z·hd powder is two containers.

made with five different bottles showed minimal variations in properties among batches.

The Brookfield Viscometer and Its Use

The viscosity of all samples was measured with a Brookfield LVT 115/60 viscometer using the LV set cylindrical spindles, and an ultra low (UL) adapter when necessary. The cylindrical spindle was placed within an outer cylindrical container with the sample in between. The inner spindle was rotated relative to the fixed outer cylinder. The torque applied to rotate the spindle, measured by a spring, is related to sample viscosity, as described below.

Cylindrical spindle geometry was chosen over "cone and plate" geometry due to the significantly wider range of shear rates and viscosities that could be measured. The UL adapter was used for low-viscosity measurements. Shear rate was varied by adjusting the rate of spindle rotation and spindle/container radii. In general, the maximum measurable shear rate decreases with sample viscosity (see vid. Table 2). We found that very low shear rates (roughly 2s^{-1} or less) were accurately measurable only with the lowest viscosity samples, when the UL adapter could be used. Instrument calibration was checked using nominally 50 and 1000 cps standards purchased from Brookfield Engineering. Brookfield claims repeatability within 1%; however, this was found to be true only with lower-viscosity Newtonian liquids at the highest spindle rotation speeds, and only if the temperature of the sample was very carefully controlled. For temperature control, the Brookfield EX 200 constant temperature bath was used. Barium sulfate sample viscosities were measured in the temperature range 23–27°C, with a few measurements at body temperature (37°C).

The Brookfield Viscometer measures viscosity through the torque required to rotate the spindle.

The spindle is driven by a synchronous motor through a calibrated spring. The deflection of the spring is visually read by a pointer, giving the percentage of the full scale torque. The average surface shear stress τ (dyne/cm²) is related to the torque T (dyne·cm) by the equation

$$\tau = \frac{T}{2\pi LR_s^2} \quad (2)$$

where R_s and L are radius and effective length (corrected for the end effects) of the cylindrical spindle, respectively (cm). The viscosity μ (poise) is defined as the ratio of shear stress τ to shear rate S (1/s) at the spindle wall*:

$$\mu = \frac{\tau}{S} \quad (3)$$

For cylindrical spindles, the Brookfield manual claims that the shear rate S depends on the angular velocity of the spindle (ω , radians/s), and the radii of the spindle and cylinder (R_s , R_c) in the following way:

$$S = \frac{2\omega}{(R_s/R_c)^2 - 1} \quad (4)$$

* With Newtonian fluids, viscosity is a constant proportionality factor between stress and twice the strain rate, both of which are represented mathematically by tensors. In the flow between concentric rotating cylinders, strain rate may be described by shear rate alone, and viscosity is the ratio of shear stress to shear rate. For the non-Newtonian fluids described in this paper, we define viscosity, μ , as the ratio between shear stress and shear rate when the fluid is subjected to shear forces. We should point out that some non-Newtonian fluids develop normal stresses in addition to shear stresses when shear forces are applied. In purely shear thinning suspensions, such as those measured in this paper, however, normal stresses are generally negligible relative to the shear stress in a shearing flow.

Table 2. Viscosity variation (μ , in cps) with temperature (T, in °C) and shear rate (S, in s^{-1}) for different mixtures, represented with least-square fitted linear curves in the range 23–27°C

Name	Shear rate (S)	Viscosity variation	Viscosity ^a (μ)	% Variation with shear rate	Measurement precision (%)
e·z·hd mix	0.07	See equation (16) and Figure 6	4803	Very large	
	6.30		358		
	12.59		298		
Liquid e·z·paque	0.12	See equation (17) and Figure 7	2261	Very large	
	6.37		318		
	12.73		227		
Syrup	0.34	$\mu = 1426.3 - 29.2T$	755	≈ 0	8.1
	0.68		7.8		
	1.35		7.3		
Mixture 1	2.52	$\mu = 5627.0 - 85.4T$	3663	9.5	4.2
	6.30	$\mu = 5854.7 - 103.2T$	3481		4.4
	12.59	$\mu = 5769.2 - 105.4T$	3345		4.1
Mixture 2	2.52	$\mu = 8585.9 - 178.2T$	4487	6.1	6.5
	6.30	$\mu = 8528.0 - 181.2T$	4360		4.9
	12.59	$\mu = 8125.1 - 169.3T$	4231		3.3
Mixture 3	2.52	$\mu = 10,893 - 243T$	5304	3.4	8.5
	6.30	$\mu = 10,862 - 243T$	5273		3.3
	12.59	$\mu = 10,766 - 245T$	5131		2.1
Mixture 4	2.52	$\mu = 12,164 - 255T$	6299	3.0	4.3
	6.30	$\mu = 12,085 - 252T$	6289		3.1
	12.59	$\mu = 11,495 - 234T$	6113		3.6
Mixture 5	1.26	$\mu = 31,220 - 802T$	12,774	7.2	8.6
	2.52	$\mu = 29,797 - 750T$	12,547		6.3
	6.30	$\mu = 26,982 - 655T$	11,917		4.0
Mixture 6	0.63	$\mu = 52,998 - 1187T$	25,697	2.3	7.1
	1.26	$\mu = 52,679 - 1171T$	25,746		5.0
	2.52	$\mu = 50,355 - 1098T$	25,101		3.3
Mixture 7	2.52	$\mu = 224,770 - 4030T$	132,080		7.6

^a Measured at room temperature ($20 \pm 1^\circ\text{C}$) for e·z·hd and liquid e·z·paque; 23°C for syrup–barium mixtures.

However, equation (4) is derived assuming a Newtonian fluid with constant viscosity μ independent of shear rate. Such a fluid is characterized by a straight line on a plot of τ vs. S (line A, Fig. 2). If fluid viscosity varies with shear rate, the use of the Newtonian relationship (eq. 4) to calculate viscosity from measured shear stress can introduce significant error [12]. This is especially true when the gap between the spindle and the container is relatively large. To overcome this difficulty, we have devised a new methodology that takes into account variations in viscosity with shear rate.

New Methodology for Measuring Viscosity Variations with Shear Rate

The degree of error introduced by using the Newtonian relationship (eq. 4) to describe non-Newtonian fluids can be derived analytically for three typ-

ical non-Newtonian fluids shown in Figure 2, curves B, C, and D. Curve B defines a fluid that requires a yields stress μ_1 before it deforms. Above μ_1 , shear stress, τ , increases linearly with shear rate, S with slope μ_0 :

$$\tau = \mu_0 S + \mu_1 \quad (\text{curve B}) \quad (5)$$

Curves C and D, on the other hand, describe “shear thickening” and “shear thinning” fluids in which viscosity increases (C) or decreases (D) with shear rate at low shear rates. These fluids may be described with a power law,

$$\tau = \mu_2 S^k \quad (\text{curve C, } k > 1; \text{ curve D, } k < 1) \quad (6)$$

up to some maximal shear rate, beyond which shear stress varies linearly with shear rate and viscosity is constant. Note that equations (5) and (6) describe Newtonian fluids when $\mu_1 = 0$ or $k = 1$, respectively.

For the non-Newtonian fluids B, C, and D, shear rate at the spindle wall is given by (see Appendix

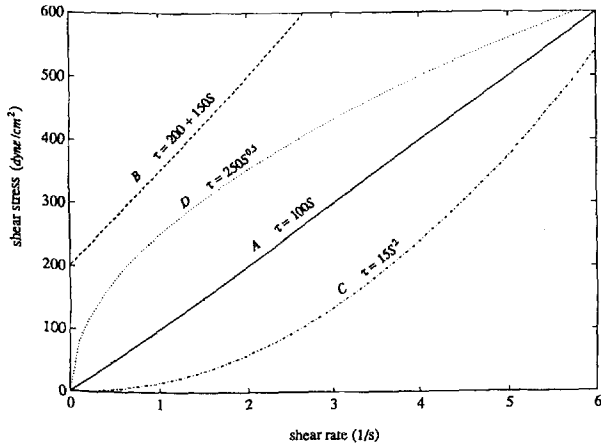


Fig. 2. Characteristics of shear stress vs. shear rate for typical non-Newtonian fluids. *A*, Newtonian fluid, $\tau = \mu_0 S$; *B*, non-Newtonian fluid with yield shear stress, $\tau = \mu_0 S + \mu_1$; *C*, shear thickening non-Newtonian fluid, $\tau = \mu_2 S^k$ ($k > 1$); *D*, shear thinning non-Newtonian fluid, $\tau = \mu_2 S^k$ ($k < 1$).

$$S = \frac{2\omega}{(R_s/R_c)^2 - 1} + \frac{\mu_1}{\mu_0} [g(R_s, R_c) - 1] \quad (\text{curve B}) \quad (7a)$$

where

$$g(R_s, R_c) = \frac{2 \ln(R_s/R_c)}{(R_s/R_c)^2 - 1} \quad (7b)$$

and

$$S = \frac{2\omega}{k[(R_s/R_c)^{2/k} - 1]} \quad (\text{curves C, D}) \quad (8)$$

Note that the Newtonian approximation introduces significant error in the estimated value for shear rate. Furthermore, at the estimated shear rate error is introduced in the viscosity estimated from measured shear stress. The relative error associated with using the Newtonian relationship (eq. 4) to calculate viscosity of non-Newtonian fluids B, C, and D from measurements of spindle rotation rate and torque is given by (see Appendix):

$$\frac{\mu_c - \mu_a}{\mu_a} = \frac{(\mu_1/\mu_0)[g(R_s, R_c) - 1]}{S + (\mu_1/\mu_0)} \quad (\text{fluid B}) \quad (9)$$

$$\frac{\mu_c - \mu_a}{\mu_a} = \left[\frac{(R_s/R_c)^2 - 1}{k[(R_s/R_c)^{2/k} - 1]} \right]^k - 1 \quad (\text{fluids C, D}) \quad (10)$$

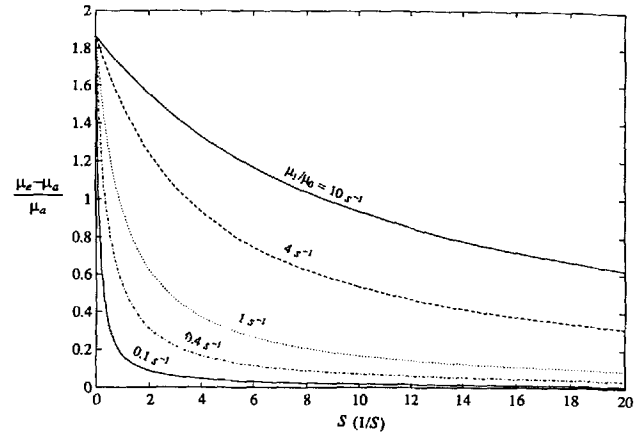


Fig. 3. Errors in viscosity estimated by using the Brookfield-suggested Newtonian method with a non-Newtonian fluid of form $\tau = \mu_0 S + \mu_1$ (curve B, in Fig. 2). The geometry of the container and spindle is that used in the current measurements ($R_s = 0.94$ cm, and $R_c = 3.54$ cm). Measured shear rate is plotted against relative error in viscosity, $(\mu_c - \mu_a)/\mu_a$, at that shear rate, according to equation (9). μ_c is the viscosity estimated by the Newtonian relationship, and μ_a is the actual or correct viscosity. The different curves in the figure are for different non-Newtonian parameters μ_1/μ_0 . As $\mu_1/\mu_0 \rightarrow 0$, the fluid becomes Newtonian.

where μ_a is the actual viscosity of the fluid at the estimated shear rate and μ_c is the viscosity estimated by using Newtonian formulas suggested by Brookfield. Figures 3 and 4 show relative error variations with non-Newtonian parameters μ_1/μ_0 or k for our often used container-spindle combination ($R_s = 0.94$ cm, $R_c = 3.54$ cm). Note that the farther μ_1/μ_0 is from 0 or k from 1, the more non-Newtonian is the fluid, and the greater the error in viscosity.

We use the following method for estimating the variation in viscosity with shear rate using either of equation (7) or equation (8), depending on which relationship provides the best fit to the measurement data. What is measured is torque T , or equivalently, surface shear stress τ (eq. 2), vs. spindle rotation rate ω . Substituting equation (7) into (5), and (8) into (6) produces the relationship between τ and ω for non-Newtonian forms considered here:

$$\tau = \mu_0' \omega + \mu_1' \quad (\text{curve B}) \quad (11)$$

where

$$\mu_0' = \mu_0 \frac{2}{(R_s/R_c)^2 - 1} \quad (12)$$

$$\mu_1' = \mu_1 \frac{2 \ln(R_s/R_c)}{(R_s/R_c)^2 - 1} \quad (13)$$

and

$$\tau = \mu_2' \omega^k \quad (\text{curves C, D}) \quad (14)$$

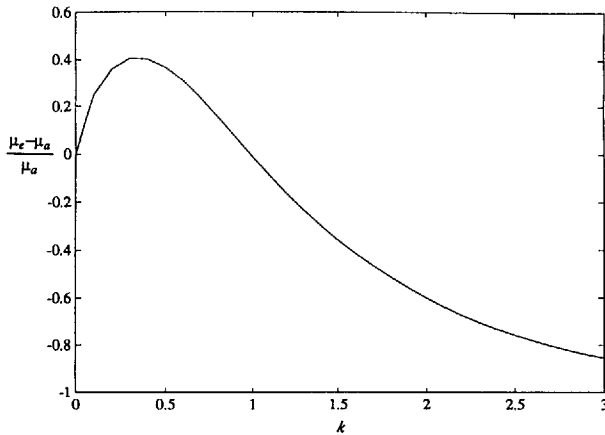


Fig. 4. Errors in viscosity estimated by using the Brookfield-suggested Newtonian method with a non-Newtonian fluid of power law form, $\tau = \mu_2 S^k$ (curves, C, D in Fig. 2). The geometry of the container and spindle is that used in the current measurements ($R_s = 0.94$ cm, and $R_c = 3.54$ cm). The relative error in viscosity, $(\mu_c - \mu_a)/\mu_a$ is plotted against non-Newtonian parameter, k , according to equation (10). μ_c is the viscosity estimated by the Newtonian relationships, and μ_a is the actual or correct viscosity. Note that in this case, the relative error is independent of shear rate. As $k \rightarrow 1$, the fluid becomes Newtonian.

where

$$\mu_2' = \mu_2 \left[\frac{2}{k[1 - (R_s/R_c)^{2/k}]} \right]^k \quad (15)$$

Note that the variation between τ and ω has the same form as the variation between τ and S . Thus, measurements of τ vs. ω directly provide the form of the non-Newtonian character of the fluid. In practice, however, measurements are made over a limited range of spindle rotation rates. One therefore uses the form of equation (11) or (14), which provides the best fit to the segment of measured data in a least-square sense. Having fit the data to equations (11) and (14) and chosen the best fit, (μ_0', μ_1') or (μ_2', k) is determined. Equations (12), (13), or (15) provide (μ_0, μ_1) or μ_2 . Shear rate is then calculated using either equation (7) or (8), and the viscosity corresponding to that shear rate is found from equation (3).

Figure 5 provides a realistic illustration of the accuracy of the method for a shear thinning fluid in comparison with the use of expressions appropriate to Newtonian fluids. The Newtonian expressions produce a large error in this case. Least-square fits of τ against ω data are, in this case, more precise using a power law representation; the predicted viscosity matches the actual viscosity. What is interesting is that piece-wise continuous fits to equation (11) also work very well in predicting the variation

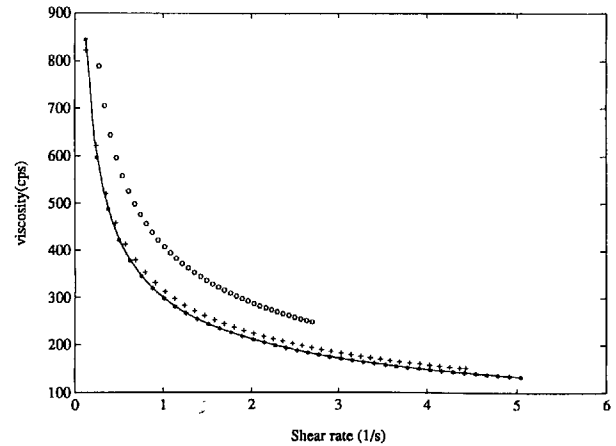


Fig. 5. Comparison between Brookfield-suggested Newtonian method and our method for estimating the viscosity of a non-Newtonian fluid. Here, the solid line is the actual viscosity given by $\mu = 300/S^{0.5}$ cps and $(R_s, R_c) = (0.94, 3.54)$ cm. ****, Viscosity estimated using the power law relationship, equation (14); + + + +, viscosity estimated using the "yield stress" relationship, equation (11); oooo, viscosity estimated using the Newtonian relationship, equation (4). Note that the Newtonian relationship introduces significant error due to the variation of viscosity with shear rate. Note also that when the "yield stress" relationship (eq. 11) is applied in piecewise segments, the accuracy is much improved over the Newtonian method, even though the fluid obeys a power law relationship.

of viscosity with shear rate, which suggests that the general methodology works well overall. Comparisons between methods are made with real data in Figures 6 and 7. Great improvements are gained over the standard Brookfield approach.

We argue that the method we propose for estimating viscosity variations with shear rate should replace the standard Brookfield method, which assumes Newtonian behavior at each spindle rotation rate. We note that the accuracy in the new method improves as the range of spindle rotation rates in which measurements are collected is increased. An important point is that the non-Newtonian character of the fluid can be deduced directly from measurements of wall shear stress against spindle rotation rate (eqs. 11 and 14). Consequently, as data are collected, one can deduce to what extent a more extended range of rotation rates is required to obtain an accurate measurement of viscosity. When viscosity has a strong dependence on shear rate, it is particularly important to measure τ against ω adequately over the range in which the non-Newtonian behavior exists. Another point to appreciate is that all methods are more accurate with smaller gap spacings (Appendix), although the method proposed

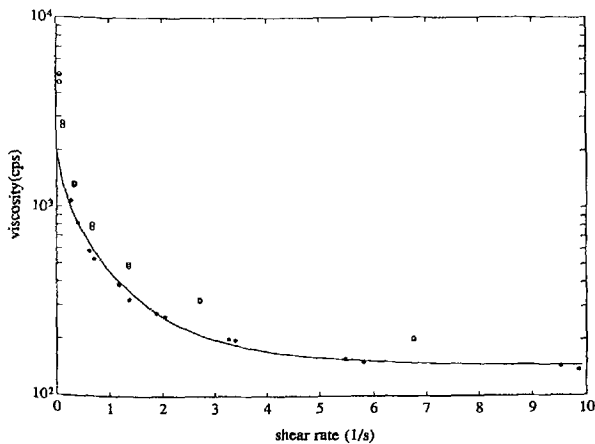


Fig. 6. Variation of viscosity with shear rate of the standard e·z·hd mixture of one container of e·z·hd powder (340 g) with 65 ml water (25% w/v). The empirical curve through the measurement data (*) is described by equation (16). For comparison, the viscosity data estimated by Brookfield suggested method are also shown by the open circles. The measurements were carried out at $20^{\circ}\text{C} \pm 1^{\circ}\text{C}$.

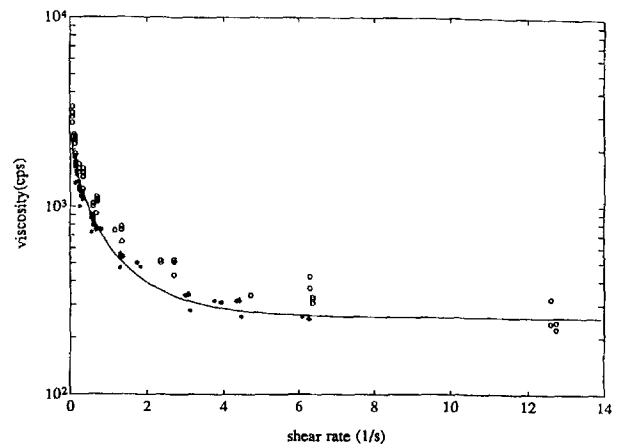


Fig. 7. Variation of viscosity with shear rate of the premixed diluted mixture of liquid e·z·paque (40% w/v). The empirical curve through the measurement data (*) is described by equation (17). For comparison, the viscosity data estimated by Brookfield suggested method are also given by the circles (o). The measurements were carried out at $20 \pm 2^{\circ}\text{C}$.

here will always provide more accurate measurements of shear-rate-dependent viscosity.

Experimental Method

In our experiments, eight rotation speeds, two sizes of Brookfield LV-set cylindrical spindles, and two sizes of Griffin beaker containers (400 ml and 600 ml) were used. For the lowest viscosity samples, the UL adapter was used. The samples were mixed uniformly without introducing air bubbles with an electric drill and a small paint-mixer attachment.

The effective length of the spindle, L , was determined through careful measurement of known standards (nominally 50 and 1000 cps). A different value of effective length was measured for each spindle-container combination. Viscosity and shear rate were calculated from measurements of torque vs. spindle rotation rate using the method described above.*

To estimate the precision of the measurements made with the syrup and syrup/barium mixtures, we repeated these tests several times (typically six times) for each combination of spindle, container, spindle speed, temperature, and fluid sample. The

average value and percentage variation for each measurement were calculated. The right-hand column of Table 2 lists the maximum percentage variation at each measured shear rate for each sample. We have found that the precision of the measurements decreases with decreasing spindle rotation rate, and the average maximum percentage variation for all viscosity measurements of syrup mixtures is about 5.8%.

Important Observations in Technique

Experience has taught us the importance of following certain procedures in mixing the samples and measuring their viscosities to minimize uncertainty:

Mixing the Sample. As will be shown, viscosity is sensitive to the ratio of e·z·hd powder to syrup. Thus, great care was taken to measure precisely the volume of syrup for given viscosity. To increase precision, two containers of e·z·hd powder and a relatively narrow graduated beaker to measure syrup volume were used. All syrup was removed from the beaker using a rubber spatula. The mixture was well stirred, and care was taken not to mix in bubbles of air (since bubbles can alter the apparent viscosity). For this purpose, we used an electric drill with a small paint-mixer attachment.

Sample Temperature. It was found to be important to measure the temperature in the center of the sample. Increasing or decreasing the temper-

* Either the Brookfield calibration for effective spindle length, or spring tension vs. torque, must be accepted by the user. We chose to accept Brookfield's spring calibration and measure effective spindle length ourselves.

ature, especially the temperature of high-viscosity samples, typically required 1 h or more without stirring (stirring reduces this time by about one-half). Great care was taken in our measurements to ensure that the sample temperatures had reached steady uniform values. For this reason, the process of data collection was very time-consuming.

Cleaning the Wall of the Spindle. It was found to be important to clean the spindle often to prevent barium from sticking to the wall and contaminating subsequent measurements.

Sample Usage Time. The viscosity of the prepared high-viscosity sample was observed to change significantly after 2 or 3 days. All measurements were made within a few hours after creation of the sample.

Measurement Results

Standard Barium Sulfate Suspensions

Variation in viscosity with shear rate was measured at room temperature ($20 \pm 1^\circ\text{C}$) for the two commercial barium sulfate mixtures listed in Table 1. Figures 6 and 7 show the rheologic characteristics of standard mixture of e·z·hd and diluted mixture of liquid e·z·paque, respectively. In both solutions viscosity is approximately independent of shear rate above about 3s^{-1} . This Newtonian behavior is especially apparent with the e·z·hd mix. Below 3s^{-1} , apparent viscosity of the suspension increases rapidly with decreasing shear rate due to the increasingly strong interactions among suspended barium sulfate particles. The curves through the data in Figures 6 and 7 are given by the empirical formulas,

$$\text{e}\cdot\text{z}\cdot\text{hd (Fig. 6): } \mu = 145 + 200e^{-1.85S^{0.6}} \text{ (cps)} \quad (16)$$

$$\text{liquid e}\cdot\text{z}\cdot\text{paque (Fig. 7):} \\ \mu = 254 + 2530e^{-1.92S^{0.6}} \text{ (cps)} \quad (17)$$

At shear rates above the rather low value of 3s^{-1} , viscosity approaches constant values of 145 cps for e·z·hd, and 254 cps for liquid e·z·paque.

The two empirical formulas in equations (16) and (17) are deduced based on the assumption of a functional form $\mu = \mu_0 + c_0e^{c_1S^k}$, where μ_0 is the viscosity in the high shear rate limit. μ_0 and k are determined using an optimization method, and parameters c_0 and c_1 are decided using extended least-square fits through the measurement data for each optimization step. Other empirical formulas in this paper are determined in a similar way.

As measures of formula precision, we define the average residual and the average percentage difference between measured and fitted viscosity values as follows:

$$\left. \begin{aligned} \text{residual} &= \left[\frac{\sum_n (\mu_{\text{formula}} - \mu_{\text{meas}})^2}{n} \right]^{1/2} \\ \% \text{ difference} &= \frac{100}{n} \sum_n \frac{|\mu_{\text{formula}} - \mu_{\text{meas}}|}{\mu_{\text{meas}}} \end{aligned} \right\} (18)$$

The average residuals of equations (16) and (17) are 37 cps and 83 cps, respectively; the percentage differences are 5.0% for equation (16) and 6.7% for equation (17). Large scatter in the data around equation (17) exists due to less precise control of temperature in the early measurements.

Viscosity of Knott's Strawberry Syrup

Because Knott's strawberry syrup is to be mixed with barium sulfate powder to increase sample viscosity, its own rheologic characteristics are of interest. Knott's strawberry syrup is Newtonian: its viscosity is independent of shear rate. There is a dependency on temperature, however, as shown in Figure 8. Viscosity decreases with temperature along the straight line given by: $\mu = 1430 - 29.2T$, where T is in $^\circ\text{C}$, and μ is in cps. In the range $23\text{--}27^\circ\text{C}$, syrup viscosity varies from 758 cps to 642 cps. Three different shear rates are plotted in Figure 8 to demonstrate the Newtonian character of the syrup.

Viscosities of Barium Sulfate Powder/Syrup Mixtures

A very large number of viscosity measurements were made of the seven mixtures of barium sulfate and Knott's strawberry syrup listed in Table 1. To the extent possible within the constraints of the instrumentation, viscosity variations with shear rate were measured. At the lower viscosities (mixtures 1, 2, 3, 4) it was possible to make measurements at shear rates between 2.5s^{-1} and 12.6s^{-1} , whereas at the higher viscosities the range of measurable shear rates reduced to $1.3\text{--}6.3\text{s}^{-1}$ for mixture 5, and $0.6\text{--}2.52\text{s}^{-1}$ for mixture 6. At all shear rates, viscosity was found to vary approximately linearly with temperature in the range $23\text{--}27^\circ\text{C}$. These data are summarized in the form of the empirical relationships in Table 2.

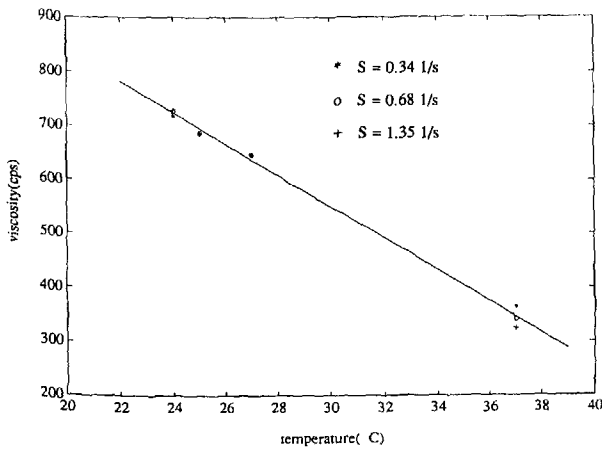


Fig. 8. The variation of viscosity with temperature at different shear rates for Knott's strawberry syrup is approximated with a straight line over the narrow range of measured temperatures.

To discern better the extent of viscosity variation with shear rate, the right-hand columns in Table 2 give the viscosities from the empirical formulas at 23°C and the percentage difference between the minimum and maximum measured shear rates. Note that the percentage difference decreases overall as sample viscosity increases, as does the minimum shear rate measurable. Within the limited range of shear rates measurable with our viscometer, the measurements indicate that the more viscous mixtures display less variation in viscosity with shear rate and begin this roughly Newtonian behavior at lower shear rates. Within experimental precision, mixtures 2 through 6 are approximately Newtonian over the range of shear rates measured, whereas the lowest-viscosity mixture, mixture 1, is slightly shear thinning. Because the value of μ_1/μ_0 is rather small in these measurements, ranging from 0.01 to 0.1, the errors in viscosity estimated by using the Brookfield-suggested Newtonian method are, in this case, within the limits of experimental precision (see Fig. 3).

It may be of interest to note that, contrary to the authors' intuitive expectations, mixtures 1–4, which are several thousand times more viscous than water, appeared surprisingly "liquid." We had expected something much thicker, perhaps closer to a "paste-like" consistency. However, it is not until viscosities on the order of 20,000–100,000 cps are reached that distinctly "paste-like" boluses are created. (Boluses in this range were readily swallowed by the second author, who is a relatively healthy person.)

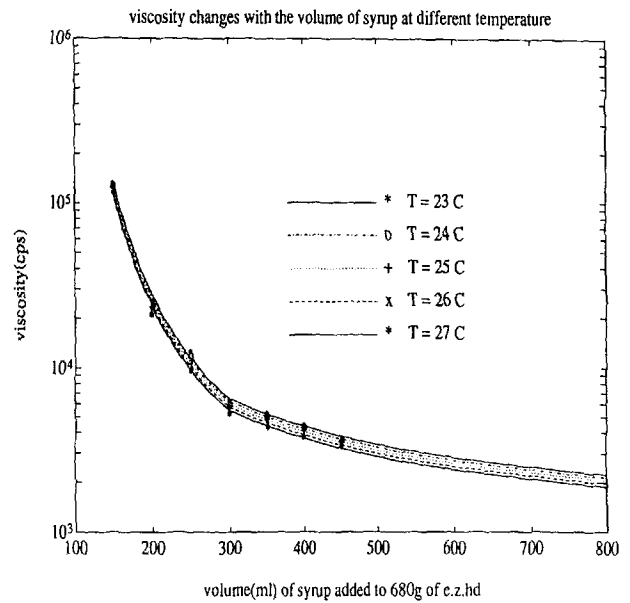


Fig. 9. Variation of viscosity with syrup to barium ratio at fixed temperatures, for shear rate $S = 2.52s^{-1}$ over the entire range of measurements. The data points are given by the symbols. The curves are given by equations (19a) and (19b), which are found by use of least-square fits. The mixture ratio is given as volume of Knott's strawberry syrup added to two containers (680 g) of e.z.hd powder. Note that the viscosity scale is logarithmic.

Empirical Relationships

In an effort to present the large amount of measured data in a form more readily usable for the development of radiopaque "test kits," the data collected at $2.52s^{-1}$ were restructured to display directly the dependence of viscosity on the ratio of Knott's strawberry syrup to e.z.hd barium sulfate powder at fixed temperature. Empirical relationships between viscosity and syrup-to-barium ratio were then derived using least-square fits over modified exponential functions at fixed temperature, in the range 23–27°C. The linear dependence of viscosity with temperature was incorporated into the formulas to provide two simple empirical relationships that fit all measured data, in the range $755 \text{ cps} < \mu < 135,000 \text{ cps}$. These relationships are given by the following equations:

$$\mu = (1430 - 29.2T)e^{3010/V_{\text{sy}}^{1.27}} \quad V_{\text{sy}} < 300 \text{ ml} \quad (19a)$$

$$\mu = (1430 - 29.2T)e^{117/V_{\text{sy}}^{0.70}} \quad V_{\text{sy}} \geq 300 \text{ ml} \quad (19b)$$

where, again, μ is in cps and T is in °C. For ease of use by the medical community, concentration of syrup to barium is given as the volume of syrup,

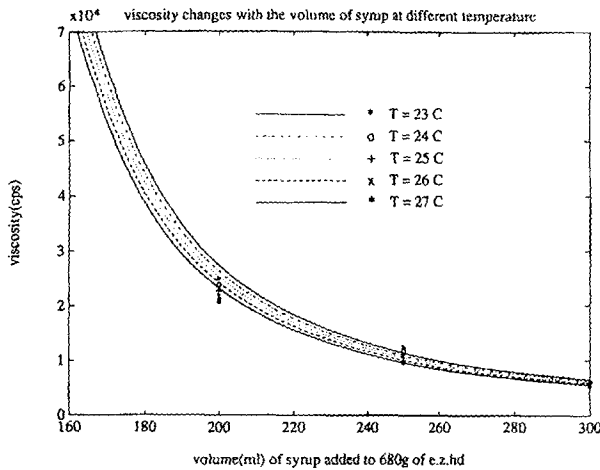


Fig. 10. Variation of viscosity with syrup to barium ratio at fixed temperatures, for shear rate $S = 2.52s^{-1}$. The data points are given by the symbols. The curves are given by equation (19a), which is found using extended least-square fits. The mixture ratio is given as volume of Knott's strawberry syrup added to two containers (680 g) of e·z·hd powder.

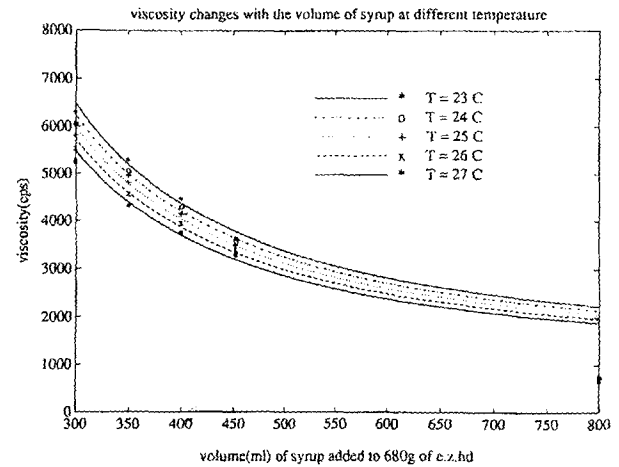


Fig. 11. Variation of viscosity with syrup to barium ratio at fixed temperatures, for shear rate $S = 2.52s^{-1}$. The data points are given by the symbols. The curves are given by equation (19b), which is found using extended least-square fits. The mixture ratio is given as volume of Knott's strawberry syrup added to two containers (680 g) of e·z·hd powder. The viscosities for pure Knott's strawberry syrup at fixed temperatures (independent of shear rate) are given by the points on the right-hand scale.

V_{syr} (ml), added to two containers (680 g) of e·z·hd barium sulfate powder. The empirical relationships are split into two formulas to improve precision. Note that as V_{syr} becomes very large the formulas yield the viscosities of syrup alone. As defined in formula (18), the average residual and average percentage difference between measured and fitted viscosity values are 1090 cps and 5.9%, respectively.

Figure 9 is a compilation over the entire range of measurements, plotted on a semilog scale. Figures 10 and 11 show the empirical curves plotted together with the data, respectively corresponding to equations (19a) and (19b). Each curve gives the variation in viscosity with volume of syrup added to 680 g (two containers) of e·z·hd at fixed temperature. Constant temperature curves between 23°C and 27°C are shown. The viscosities of syrup alone (758–642 cps in the temperature range 23°–27°C) are given by the data points on the right-most edge of Figure 11. Viscosity increases with decreasing syrup to barium ratio. However, note that this increase in viscosity is more rapid as the syrup to barium ratio decreases. When V_{syr} is below about 300 ml, viscosity is especially sensitive to syrup to barium ratio, as is evident in Figure 9.

Bolus Density

As was discussed previously, viscosity is associated with the frictional forces within the bolus fluid that resist attempts to propel the bolus forward, or keep the bolus moving. However, even in the absence of

friction, force must be applied to initiate bolus motion or to change the bolus velocity. This applied force, required to overcome the inertia of the fluid, is proportional to fluid density.* Furthermore, the gravitational force (bolus weight) is proportional to the bolus density. Gravity is important when the subject is in a nearly vertical position and when bolus viscosity is sufficiently low that frictional forces are small relative to gravitational forces.

Fluid inertia is not high during esophageal bolus transport due to low peristaltic wave speeds. If radiography is carried out with minimal effects of gravity (e.g., the subject in a prone position), changes in bolus density are not expected to affect bolus transport significantly. Thus, viscosity is the primary rheologic variable for esophageal study. However, during the pharyngeal phase of bolus transport, fluid density can be expected to play a significant role. This is especially true during the initiation of the swallow, when the bolus is rapidly accelerated as it is propelled into the pharynx, and during the initial period of the UES opening, as the bolus makes contact with the upper esophageal sphincter and rapidly decelerates [13].

* Note that "specific gravity" is defined as the ratio of the density of the fluid to that of water, 1 g/ml at room temperature.

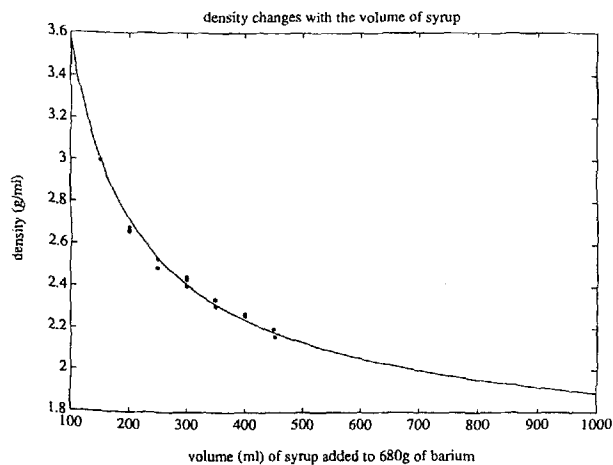


Fig. 12. Variation of density with syrup to barium ratio at room temperature. The mixture ratio is given as volume of Knott's strawberry syrup added to two containers (680 g) of e·z·hd powder.

Unfortunately, it was not possible to differentiate changes in mixture viscosity from changes in mixture density. To begin with, the density of Knott's strawberry syrup is 1.41 times that of water, so the density of the standard e·z·hd mixture, 1.8 g/ml, is significantly less than the syrup-based higher viscosity mixtures in Table 1. Density variation as a function of syrup to barium ratio is shown in Figure 12, where density is plotted against the volume (ml) of syrup added to two containers of e·z·hd powder. The data are fitted with the following empirical equation:

$$\text{density (g/ml)} = 1.41e^{9.32/\sqrt{V_{\text{syr}}}} \quad (20)$$

A 20% variation in density occurs over the range 6000–60,000 cps. According to equation (18), the average residual and average percentage differences between measured and fitted density values are 0.03 (g/ml) and 1.0%, respectively.

Suggested Test Kit for Motility Studies

For the radiographic study of esophageal and pharyngeal motility and for clinical analysis of motility disorders that are bolus-dependent, we suggest the systematic use of a test kit of barium mixtures with well-defined rheologic characteristics. In particular, we suggest the use of at least two, but preferably more, bolus viscosities that encompass the range "liquid" to "pasty." To cover this range adequately, we suggest three boluses that are a factor of 10 different in viscosity from each other. We propose the following standard test kit of three mixtures for repeated use in biological trials:

Low-Viscosity Sample: $\mu \sim 150$ cps

Mixture. Standard e·z·hd mix (250% w/v): one container e·z·hd powder (340 g) to 65 ml water.

Viscosity. $\mu \approx 150$ cps at $20 \pm 1^\circ\text{C}$ for shear rates, $S \geq 3\text{s}^{-1}$. Apparent viscosity increases with decreasing S below approximately 3s^{-1} .

Density. 1.8 g/ml (specific gravity = 1.8).

Mid-Viscosity Sample: $\mu \sim 6500$ cps

Mixture. Knott's strawberry syrup 300 ml with two containers e·z·hd powder (680 g).

Viscosity. $\mu \approx 6480$ cps at 23°C for shear rate $S = 2.5\text{s}^{-1}$. Apparent viscosity may decrease slightly with increasing S above 2s^{-1} .

Density. 2.4 g/ml (specific gravity = 2.4).

High-Viscosity Sample: $\mu \sim 63,000$ cps

Mixture. Knott's strawberry syrup 170 ml with two containers e·z·hd powder (680 g).

Viscosity. $\mu \approx 63,000$ cps at 23°C for shear rate $S = 2.5\text{s}^{-1}$. Apparent viscosity does not change significantly above 0.6s^{-1} .

Density. 2.9 g/ml (specific gravity = 2.9).

Preparation and Use of Barium Samples

Great care should be taken to obtain a precise mix of syrup and barium powder. Small variations in syrup volume can cause large variations in viscosity; this is especially true at the highest viscosities (Fig. 10). Therefore, the volume of syrup should be measured carefully, and the beaker used to measure the syrup should be scraped clean with a rubber spatula. Although the mid- and high-viscosity samples can be obtained by adding half the indicated amount of syrup to one container e·z·hd powder, it is suggested that the mix be made with two containers of barium to reduce uncertainty in the mixture ratio.

Samples should be mixed and used at room temperature ($22\text{--}24^\circ\text{C}$). The temperature should be measured from the center of the sample, and should be noted. If necessary, the sample should be allowed to come to room temperature. Allowing the sample to sit for an hour, and stirring it every now and then, is probably sufficient.

Samples can be mixed ahead of time for later use. However, it is suggested that the samples be

used within 24 h after being mixed. Significant changes in viscosity were observed after a few days.

Conclusion

We have provided in this paper data and convenient empirical formulas for use in motility studies of the swallowing process. We consider that this first complete set of data directed at systematic variation in bolus viscosity should provide a basis for future studies of pharyngeal and esophageal motility in which radiography and manometry are used as primary analytical tools.

We have also provided a new methodology for measuring the relationship between viscosity and shear rate in conventional concentric cylinder-type viscometers. Standard methods based on Newtonian relationships are shown to be inaccurate when the fluid is significantly non-Newtonian. The new method greatly improves the accuracy of viscosity measurements when the dominant non-Newtonian characteristic is the dependence of viscosity on shear rate.

Mixtures other than those quantified here are certainly possible, and, indeed, should be developed and quantified. It would be of value in pharyngeal studies, for example, if bolus density and viscosity could be varied independently. A test kit of bolus substances in which other rheologic characteristics—elasticity, for example—are included, would be of great clinical value. With this study, we hope to encourage the quantification of bolus characteristics, the systematic variation of these characteristics in motility studies, and the reporting of rheologic bolus characteristics as standard practice in the study and clinical evaluation of the swallowing process.

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Appendix

Here we derive the expressions for wall shear rate with non-Newtonian fluids characterized by $\tau = \mu_0 S + \mu_1$ (eq. 5) and $\tau = \mu_2 S^k$ (eq. 6), and we evaluate

the error associated with the Newtonian-based Brookfield formulas.

The momentum equation for steady flow between two coaxial concentric cylinders reduces to the following form in cylindrical polar coordinates (r, θ) :

$$\frac{1}{r^2} \frac{\partial}{\partial r} (r^2 \tau) = 0 \quad (\text{A-1})$$

with the following boundary conditions:

$$\left. \begin{array}{l} r = R_s, \quad u_\theta = R_s \omega \\ r = R_c, \quad u_\theta = 0 \end{array} \right\} \quad (\text{A-2})$$

where u_θ is the velocity in θ direction. The relationship between shear rate and u_θ is

$$S = r \frac{\partial}{\partial r} \left(\frac{u_\theta}{r} \right). \quad (\text{A-3})$$

Let

$$\begin{aligned} \tau &= \mu_0 S + \mu_1 \equiv \mu S \\ &= \mu_0 r \frac{\partial}{\partial r} \left(\frac{u_\theta}{r} \right) + \mu_1 \end{aligned} \quad (\text{A-4})$$

Thus, “viscosity” μ varies with shear rate S ,

$$\mu = \mu_0 + \frac{\mu_1}{S} \quad (\text{A-5})$$

Substituting (A-4) into (A-1), integrating twice, and applying the boundary conditions (A-2) yields

$$\begin{aligned} u_\theta &= \frac{\omega R_s^2}{R_s^2 - R_c^2} \left(r - \frac{R_c^2}{r} \right) + \frac{\mu_1}{\mu_0} \\ &\times \left[\frac{r^2 R_s^2 \log(R_s/r) + r^2 R_c^2 \log(r/R_c) + R_s^2 R_c^2 \log(R_c/R_s)}{r(R_s^2 - R_c^2)} \right] \end{aligned} \quad (\text{A-6})$$

The shear rate on the spindle wall, equation (7), is found by substituting (A-6) into (A-3), taking the appropriate derivatives, and evaluating at $r = R_s$:

$$S = \frac{2\omega}{(R_s/R_c)^2 - 1} + \frac{\mu_1}{\mu_0} [g(R_s, R_c) - 1] \quad (\text{curve B}) \quad (7a)$$

where

$$g(R_s, R_c) = \frac{2 \ln(R_s/R_c)}{(R_s/R_c)^2 - 1} \quad (7b)$$

Note that the first term in equation (7a) results from an assumption that the viscosity μ does not vary with shear rate S (eq. 4). If we call S_a the actual,

or correct, viscosity and S_e the viscosity estimated assuming a Newtonian fluid, then

$$S_a = S_e + E_s \quad (\text{A-7})$$

where

$$E_s = \frac{\mu_1}{\mu_0} [g(R_s, R_c) - 1] \quad (\text{A-8})$$

E_s is the error made in calculating the shear rate from spindle rotation rate if one ignores the variation of viscosity with shear rate. Furthermore, at the estimated shear rate, S_e , an error is also made in the estimated viscosity μ_e . To evaluate this error, note that Brookfield estimates the viscosity μ_e from

$$\tau_m = \mu_e S_e \quad (\text{A-9})$$

where τ_m is the actual *measured* shear stress at the spindle wall. The correct viscosity μ_a at $S = S_e$ is

$$\mu_a = \mu_0 + \frac{\mu_1}{S_e} = \frac{\mu_0 S_e + \mu_1}{S_e} \quad (\text{A-10})$$

The actual *measured* value of shear stress τ_m , however, is related to μ_0 and μ_1 through the *actual* value of shear rate, S_a :

$$\tau_m = \mu_0 S_a + \mu_1 \quad (\text{A-11})$$

Equating (A-11) and (A-9) implies that

$$\mu_e = \frac{\mu_0 S_a + \mu_1}{S_e} = \frac{\mu_0 (S_e + E_s) + \mu_1}{S_e} \quad (\text{A-12})$$

Thus, the relative error in viscosity at $S = S_e$, from (A-10) and (A-12), is given by equation (9):

$$\begin{aligned} \frac{\mu_e - \mu_a}{\mu_a} &= \frac{\mu_e}{\mu_a} - 1 = \frac{\mu_0 (S_e + E_s) + \mu_1}{\mu_0 S_e + \mu_1} - 1 \\ &= \frac{E_s}{S_e + (\mu_1/\mu_0)} \\ &= \frac{(\mu_1/\mu_0)[g(R_s, R_c) - 1]}{S_e + (\mu_1/\mu_0)} \quad (9) \end{aligned}$$

The error diminishes as the ratio μ_1/μ_0 becomes very small: the fluid becomes Newtonian. In addition, it is easy to show that $g(R_s, R_c) \rightarrow 1$ as $R_c \rightarrow R_s$, so that the relative error may also be reduced by making the relative gap between the inner and outer cylinders very small. In practice, however, this is only feasible with very-low-viscosity samples (when the UL adapter can be used).

A similar procedure to the one described above may be followed to evaluate the characteristics of a power law fluid (curve C, D in Fig. 2), given by,

$$\tau = \mu_2 S^k \equiv \mu S, \quad \mu = \mu_2 S^{k-1} \quad (\text{A-13})$$

The result is

$$S = \frac{2\omega}{[k(R_s/R_c)^{2/k} - 1]} \quad (8)$$

The Newtonian expression is given by $k \rightarrow 1$. The measured shear stress τ_m is related to the estimated viscosity μ_e through the estimated shear stress S_e using (A-9), while the actual viscosity at S_e is given by

$$\mu_a = \mu_2 S_e^{k-1} \quad (\text{A-14})$$

On the other hand, the actual measured shear stress τ_m is related to μ_2 through the actual value of the shear rate, S_a by

$$\tau_m = \mu_2 S_a^k (= \mu_e S_e) \quad (\text{A-15})$$

Combining (A-9) and (A-15) with (A-14) yields the relative error in viscosity at $S = S_e$:

$$\frac{\mu_e - \mu_a}{\mu_a} = \left[\frac{(R_s/R_c)^2 - 1}{[k(R_s/R_c)^{2/k} - 1]} \right]^k - 1 \quad (\text{fluids C, D}) \quad (10)$$

As $k \rightarrow 1$ (the Newtonian fluid), the error clearly diminishes to zero. Similar to the above, error also diminishes as $R_c \rightarrow R_s$.

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