Adaptation of Commercial Viscometers for Special Applications in Pharmaceutical Rheology I

The Brookfield Viscometer

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The adaptation of the Brookfield LVT and RVT viscometers to absolute rheometers by the use of a fixed cup and bob system is described. The resultant instruments, providing shear rates in the 0.1 to 15 seconds^{-1} range, supplement the Drage Epprecht rheometer in the next two lower decades of shear. The converted LVT was used to evaluate the apparent shears found with the conventional disk spindles of the Brookfield RVF. It is shown that the resultant shear is an integration of the Newtonian and non-Newtonian components. Thus, although the use of several speeds with such spindles does give indications of pseudoplastic behavior, the shear dependency is not fully characterized.

One of the most widely available viscometers for general control use has been the Brookfield Synchro-Electric viscometer in one of its several models. For routine control measurement, it is frequently customary to use only the most suitable speed for measurement with a given spindle. Most spindles are of the disk type rather than the bob or rod. As a result of this general type of usage, the instrument has tended to be somewhat in disrepute among practicing rheologists. There have been several attempts (1–3) to apply the use of the several available speeds of the instrument, four in the F models and eight in the T models, to the study of non-Newtonian systems. However, these have been confined to the use of the normally supplied Brookfield disk spindles, and hence have been limited by the nondefinable shear-rates used.

Since this instrument in the T model has very slow speeds available, it seemed desirable to attempt to fit it with a series of cups and bobs such that reasonably absolute shear measurements might be made in the low shear-rate range.

EXPERIMENTAL

Initially a Brookfield model LVT viscometer was equipped with cups A and B of internal diameters 2.54 and 0.852 cm., respectively, attached rigidly to the viscometer by a twist plate identical to that normally used to mount the guard ring. (See Fig. 1.) The Nos. 1 and 4 spindles as normally supplied are straight geometric cylinders. Similar geometric spindles are available for Nos. 2 and 3 on order from Brookfield Engineering Corporation, Stoughton, Mass. The pertinent dimensions are given in Table I. All are flat bottomed and have a shaft diameter of 3.0 mm.

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It was intended that the use of this instrument would be primarily to complement the Drage Epprecht rheometer by supplying the lower shear-rate range. For this reason Newtonian shear rates were calculated in accordance with the standard

TABLE I.—DIMENSIONS OF CYLINDRICAL BROOKFIELD LVT SPINDLES

Dimension	Brookfield Spindle Number 1	2	3	4
Diameter, cm.	1.9	1.0	0.6	0.3
Length, cm.	6.5	5.4	4.3	3.1

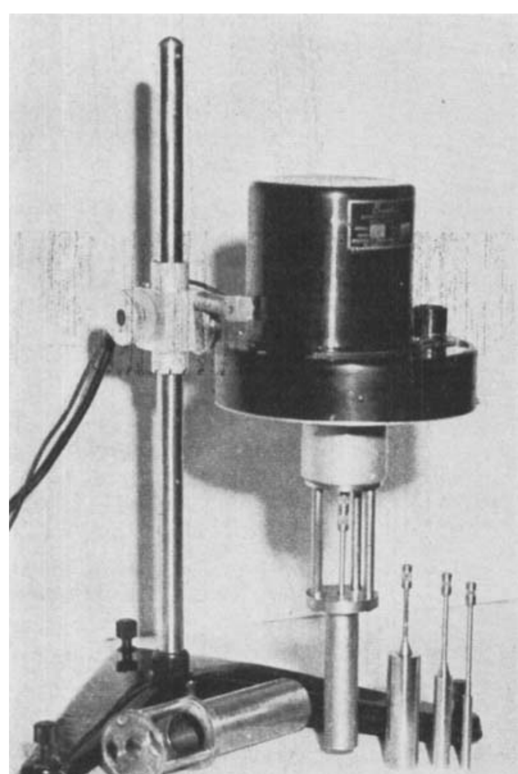

Fig. 1.—Brookfield LVT with constructed cups and cylindrical spindles.

practice for that instrument (4). The torque factor was calibrated experimentally from the slope of the plot of the scale reading of the instrument *vs.* calculated shear rates for all suitable National Bureau of Standards calibration oils. These oils are all Newtonian in the range of shears used here, so that straight line plots were obtained for each bob for each oil used. This method previously had provided torque factors for the Drage identical to those cited by the manufacturer.

Since the spindle mounting of the Brookfield is not rigid but quite free-floating, close cup-to-bob clearances are not practical. In types of non-Newtonians with which the instrument has been used for the last 3 years, the use of the *A* cup with spindles 1 and 2, occasionally 3, has been the general rule. For those systems for which the two thinnest bobs, 3 and 4, are needed it is often not practical to fill the smaller cup without the use of excessive disturbing shear of the sample. We intend to extend our usage of the smaller bobs by the insertion into the filled *A* cup of sleeves similar in design to those now available with the Drage Epprecht rheometer. These reduce the cup-to-bob ratio, and incidentally raise the shear rate used. These sleeves can be inserted into filled cups with very little shear disturbance.

Most applications of this instrument, as with the Drage, involve direct comparisons between samples measured in identical manners. This is particularly true of its use in routine aging comparisons.

For heavier consistency systems we have similarly adapted a Brookfield model RVT using the same bobs and the large cup only. Here the cup is mounted by an adapter plate which connects by the mounting screws normally used to bolt on the guard ring. The relative spring moments of the LVT and RVT models as given by the manufacturer are 6.737 and 71.97 dyne cm./division, respectively.

Apparent Shear with Regular Brookfield RVF Spindles.—Frequently it has been desirable to know under what equivalent shear routine control type Brookfield RVF readings were being taken. Accordingly six pseudoplastic materials were chosen of varying viscosity characteristics as shown in Table II.

TABLE II.—VISCOSITY CHARACTERISTIC OF TEST SUBSTANCES AS FUNCTION OF SHEAR RATE

Test Substance	Apparent Viscosity D = 0.1 sec.$^{-1}$	(Point to Origin) D = 1 sec.$^{-1}$	Cps. D = 10 sec.$^{-1}$
1	36,000	14,000	2,200
2	42,000	17,000	3,700
3	30,000	5,000	940
4	22,000	3,900	870
5	13,000	4,400	920
6	66,000	14,000	3,500

All RVF spindles which would give readings were also used with each lotion. The multiplying factors appropriate to each spindle as supplied by the manufacturer were used to calculate apparent viscosities. Plots were then made for each liquid against the relative Newtonian apparent shear rate. An example is shown in Fig. 2. The reference point chosen was the No. 7 spindle at 2 r.p.m. This has the highest multiplying factor, 20,000. This was given a relative Brookfield shear rate of 1*X*. The relative shear rates were assumed to vary with the multiplying factor. Thus spindle No. 3 at 20 r.p.m. with a multiplying factor of 50 was given a relative shear of 20,000/50 or 4000*X*. The shear rate from the rheogram necessary to give an apparent viscosity equivalent to that observed using the Brookfield multiplying factor was taken as the Spindle Equivalent Shear Rate for that operating condition.

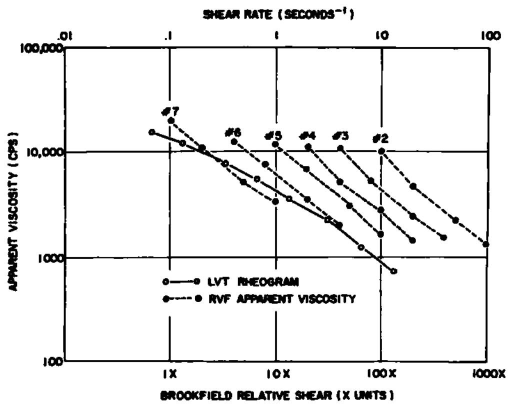

Fig. 2.—Comparison between conventional Brookfield RVF readings at each speed for the various spindles and a rheogram from the modified LVT for the same pseudoplastic lotion.

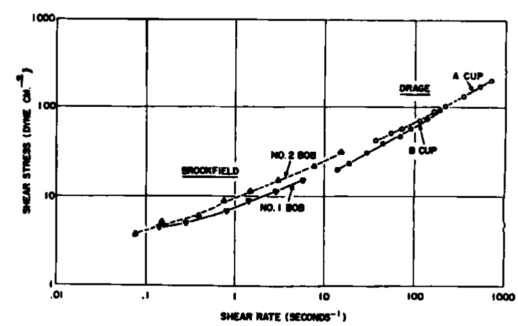

Fig. 3.—Rheograms run with the modified Brookfield LVT and with the Drage Epprecht viscometer for a pseudoplastic lotion.

RESULTS AND DISCUSSION

Rheology.—In the calculation of shear rates for cup-and-bob combinations where the radius ratio departs significantly from unity, the choice of the method of calculation is a major problem. Fischer (5) and Andrade (6) have tried different approaches to the integration of the average shear rate. Since the Newtonian viscosity is independent of the integration procedure used, the shear stress term must bear the same averaging method. It was therefore decided to take the direct approach, namely, to use the simple Newtonian shear-rate formula and, by the empirical calibration, include any necessary correction factor into the torque term.

This is justified because a multispeed instrument is of no value for Newtonian systems, but is of particular need for pseudoplastic ones. In such systems, having as they do exponential torque-shear rate dependencies, it is no longer possible to simply relate the shear gradient to the system geometry since the nature of the shear dependency enters into

TABLE III.—APPARENT SPINDLE EQUIVALENT SHEAR RATES FOR BROOKFIELD RVF SEC.$^{-1}$

Spindle Speed, r.p.m.	Brookfield Spindle Number 7	6	5	4	3	2
2	0.03–0.22	0.11–0.33	0.09–0.50	0.10–0.50	0.11–0.54	0.19–0.60
4	0.12–0.40	0.09–0.70	0.5 –1.1	0.5 –1.1	0.7 –1.2	0.8 –1.2
10	0.6 –1.4	1.3 –2.5	1.9 –2.9	2.1 –2.8	2.6 –3.2	3.2 –3.6
20	1.5 –3.8	3.7 –4.8	4.3 –5.3	4.3 –5.6	4.4 –6.2	6.0 –7.4

the observed gradient. In this case, for absolute measurements, all calculations must be reduced to the walls of the measuring system (7). In the extrapolated limit, any equations should lead to the same values for apparent shear rate and torque at the wall. It is intended that such a treatment be the subject of a later paper.

Figure 3 shows the results obtained for the measurement of an antiperspirant lotion at 25° using the Nos. 1 and 2 bobs of the Brookfield and the *A* and *B* cups of the Drage rheometer. It is apparent that this is a pseudoplastic system. The variety of cup and bob ratios used here partially justifies Fischer's statement (5) that "For non-Newtonian liquids . . . the mean value of η is found experimentally to be approximately the same for large and small clearances, so that the effect of variable rate of shear apparently balances out."

The Brookfield LVT modified in the manner shown compliments the Drage Epprecht viscometer by providing for the two to three decades of shear lower than otherwise available. This modification thus provides an economical low-shear rheometer suitable for measurements in the range normally found for tipping and slow pouring from a bottle of rather heavy lotions (8). Thus the rheological study of their aging in a useful shear rate range is facilitated. The use of this instrument in the study of Veegum suspensions is given elsewhere (9).

Apparent Shear of Brookfield RVF Spindles.—Table III gives the ranges found for the Equivalent Shear Rates for the various spindles in use with the Brookfield RVF. It will be noted in Fig. 2, that with the exception of the No. 7 spindle (which has no disk) the apparent viscosity at each speed was surprisingly independent of the bob used to measure it. (Note the correspondence of the serial spots for each bob.)

The slopes of the bob lines are relatively parallel, but do not in all cases parallel the shear-dependency line of the rheogram. No absolute correlation can be made between apparent Brookfield readings for a given speed and the viscosity of a non-Newtonian. Nevertheless, the ranges of apparent shear rate are narrow enough that we consider we can predict satisfactorily from the rheogram of a given system what its apparent Brookfield viscosity will be.

Thus readings obtained by a conventional Brookfield RVF can be empirically equated to the values found from a conventional rheogram. However, the apparent viscosity value does depend on the spindle used, as does the apparent shear dependency. The fact that such correlation is possible obviously follows from the reasonable success the instrument has had in process monitoring.

With such a low-shear rheometer, equivalent types of calibrations may be made of shear rates of other nondefinable measuring systems.

SUMMARY

A readily constructed attachment to convert a Brookfield viscometer of the T type to an absolute rheometer is described. Its application in the case of the Brookfield model LVT to give a rheometer of shear rate range 0.1 to 15 sec.$^{-1}$ is described.

The equivalent shear rate obtained by conventional Brookfield RVF with disk spindles was determined by equating the viscosities obtained to the rheogram. It is found that, since the spindle integrates the non-Newtonian with the Newtonian component, the use of the multispeed determination, as described in the literature, gives a useful but an incomplete idea of the non-Newtonian character.

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