Sensory Testing of Spreadability: Investigation of Rheological Conditions Operative during Application of Topical Preparations

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Abstract ☐ The rheological conditions which operate during spreading of topical preparations on the skin were investigated for a series of lipophilic preparations ranging from stiff semisolids to mobile liquids. Rates of shear were found to vary from approximately 400 to 2500 sec.$^{-1}$ depending on the consistency of the preparation being spread; a rheological master curve was determined. Three types of scaling procedures (ordinal, preference, and ratio scaling), employed to investigate the ability of a panel to differentiate levels of the textural parameter spreadability, were found to be of similar efficiency. The data derived were used to indicate a method for determining instrumental rheological conditions for use in routine industrial control procedures and a spreadability screening test for application during innovative work prior to field trials.

Keyphrases ☐ Topical formulations—spreadability, rheological conditions ☐ Rheological master curve—shear rate variation, spreadability of lipophilic topical preparations ☐ Spreadability of lipophilic topical preparations—sensory tests, rheology ☐ Textural profile of topical preparations—sensory tests, rheology ☐ Sensory scaling procedures—ordinal, preference, ratio

Product development studies of pharmaceutical and cosmetic preparations may culminate in extensive and time-consuming field trials in which textural properties as well as therapeutic or cosmetic properties of a product are assessed using a representative cross section of the population. While the therapeutic properties of a product may be screened using pharmacological techniques prior to such a trial, no satisfactory methods are available for preliminary screening of textural characteristics. Savings may be made in time and money if field trials are restricted to products that pass more comprehensive screening tests in the laboratory.

The consumer acceptance of a preparation for topical application, therapeutic and cosmetic effects apart, is governed by various properties of the preparation, collectively known as the textural profile, such as appearance, odor, extrudability where applicable, initial sensations upon contact with the skin, spreading properties, tackiness, and residual greasiness after application. Rheological techniques may be used to study the conditions operating during the application of a preparation to the skin. The topical application procedure may be subdivided into four sections: (*a*) removal of sample from container, (*b*) initial sensations on the skin, (*c*) sensations during spreading on the skin, and (*d*) final impressions due to residue on the skin.

Section (*a*) was investigated by Van Ooteghem (1) and Langenbucher and Lange (2); Section (*d*) sensations depend, for example, upon whether the material is oily or aqueous. The textural properties corresponding to Sections (*b*) and (*c*) are consistency and spreadability, respectively.

Consistency is assessed at low rates of shear and has been correlated with yield stresses (initial shear stresses) and with viscoelastic properties determined in creep (3–5). Spreadability is subjectively assessed at higher rates of shear. Reported data indicate that rates of shear during spreading may vary from 10^2 to 10^5 sec.$^{-1}$ (1–4, 6, 7). The rate of shear during spreading, $\dot{\gamma}$ sec.$^{-1}$, is calculated using the equation for plane laminar flow between two parallel plates:

$$\dot{\gamma} = V/d \quad \text{(Eq. 1)}$$

where V is the relative velocity of the plates (cm. sec.$^{-1}$), and d is the distance between them (cm.). Visual estimates of V may be made, although with difficulty; cinematographic techniques could be of use for more accurate work. The distance, d, between the plates (thickness of film between skin surfaces) must be estimated and varies as the preparation is spread onto the skin. It is because of difficulty in measuring d that shear rate conditions during topical application are not properly known.

Due to the lack of information concerning the rheological conditions which occur during topical application, previous investigations (and also routine industrial product control procedures) utilized largely arbitrary instrumental shear rate conditions to determine the rheological characteristics of a preparation. They then attempted to find correlations between instrumental data and sensory data. For example, in recent work (3, 4) a viscometer with discontinuous shear rate variation was used to determine the rheograms for several preparations. Various parameters, such as plastic viscosity and extrapolated yield stress, were derived from the hysteresis loops and compared with the sensory data to find the best correlations. Langenbucher and Lange (2) did attempt to determine optimum specifications for topical preparations using Newtonian oils in extrusion and spreadability tests. The disadvantage of this type of work is that most materials for topical application are not Newtonian so direct comparison is difficult. Wood (8) developed a method involving comparison of Newtonian with pseudoplastic liquids to determine the approximate rate of shear de-

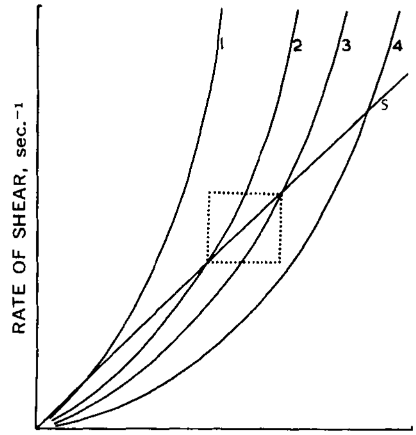

Figure 1—*Comparison of consistency of non-Newtonian materials 1–4 with the Newtonian fluid, S, in the mouth. Materials 2 and 3 were found to be closest in consistency to S [after Wood (8)].*

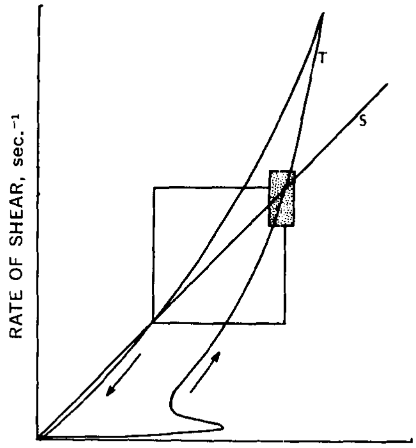

Figure 2—*Continuous shear flow curves (Instrumental Method 1) for a Newtonian fluid, S, and a time-dependent shear thinning semisolid product, T. The open rectangle indicates approximate rheological conditions concerned with the total intersection area; the shaded rectangle indicates conditions localized around up curve of the T rheogram.*

veloped in the mouth. This method may be adapted for investigation of materials for topical application and has the advantage that the velocity, V, and the film thickness, d, need not be known to determine the shear stress/shear rate conditions which occur during spreading.

In the present work, this method is modified and extended to investigate the rheological conditions which operate during spreading of lipophilic emulsions and pastes on the skin. The preparations employed cover the range of consistency from mobile liquids to stiff semisolids, so that any variation in the rheological conditions dependent upon the consistency may be determined. The spreadability of the preparations is investigated using different types of scaling procedures.

Both arithmetic and logarithmic plots of data are employed in this work; to avoid confusion between the two types of plot, the former have shear rate on the Y-axis and shear stress on the X-axis while the latter are the inverse of this system.

THEORY

Rheological Conditions during Spreading—Wood (8) developed a method for evaluating the shear stress/rate of shear conditions which occur in the mouth during evaluation of the consistency of a liquid foodstuff. Pseudoplastic liquids (soups) were assessed by a panel whose members were asked to say which soup was nearest in consistency to a given Newtonian fluid. The intersection of the flow curves of the selected soup(s) and the standard fluid indicated the approximate shearing conditions which occurred during the assessment (Fig. 1). The rectangle in Fig. 1 indicates the approximate rheological conditions, assuming Samples 2 and 3 are selected by a panel as being most similar to the Newtonian standard. This method is easily adapted for investigation of spreading of topical preparations.

One difficulty is that rheograms of such preparations are often hysteresis loops because of time-dependent structural breakdown during shear. In some circumstances, the intersection of a Newtonian fluid with such a hysteresis loop may cover a wide range of shearing conditions (shown by the open rectangle in Fig. 2). However, it is unlikely that the down curve of the rheogram will correlate with any sensory parameter because it represents the rheological characteristics of a material after prolonged shearing deformation. Such a process would not normally occur during topical application because consumer evaluation of viscosity or spreadability probably occurs quickly, and longer times may not be relevant. The actual shear conditions may, therefore, be assumed to be localized around the intersection of the flow curve for the Newtonian fluid and the up curve of the test material rheogram (shown by the shaded rectangle in Fig. 2).

Several factors must be considered when obtaining the rheograms. A thin film of material spread on the skin rapidly warms to the skin temperature. The skin temperature may vary during spreading of a topical preparation due to increased blood flow induced by rubbing. Rheological work for correlation with spreadability must, therefore, be performed at a suitable temperature. The time scale of instrumental experiments is also important. When a consumer applies a topical preparation, the spreading procedure is of relatively short duration. Rheograms should be such that the (assumed) operative rate of shear is attained in a time comparable with the "time of spreading" of a topical preparation by a consumer. The time of spreading is a qualitative guide only as it may contain several time components, each of which is associated with a different parameter of the textural profile (*e.g.*, stickiness).

The Wood (8) method was therefore modified. It is possible to measure the shearing stress of a test material at a constant rate of shear (*a*) immediately after application of the shear rate and (*b*) after a given time which corresponds to the time of spreading. The process is repeated at various shear rates, and the data are plotted on double logarithmic axes as shown in Fig. 3. The shear conditions during spreading are located approximately by the intersection of the test data curves and the flow curve for a Newtonian fluid judged by a panel to be of similar spreadability. Alternatively, an automatic viscometer with a continuously variable rate of shear may be employed to record a rheogram over a suitable time scale; the shear

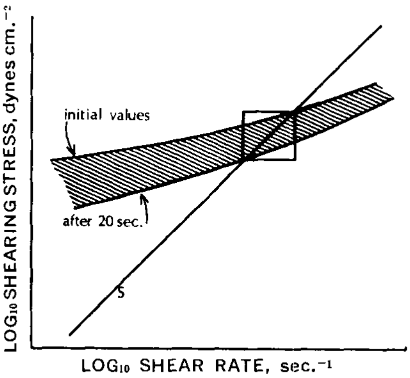

Figure 3—*Discontinuous shear flow curves (Instrumental Method 2) for a Newtonian fluid, S, and a time-dependent shear thinning semisolid product, T. The rectangle indicates approximate rheological conditions in the region of the intersection of the curves.*

Table I—Formulations of Preparations Used

Sample Number	White Soft Paraffin	Hard Paraffin	Liquid Paraffin	Light Liquid Paraffin	Wool Fat	Cetostearyl Alcohol	Iso-propyl Myris-tate	Starch	Zinc Oxide	Sorbitan Monooleate	Water
1	50	—	—	—	—	—	—	26	24	—	—
2	50	—	—	—	—	—	—	50	—	—	—
3	70	—	—	—	—	—	—	30	—	—	—
4	90	—	—	—	—	—	—	10	—	—	—
5[a]	42.5	2.5	—	—	2.5	2.5	—	—	—	0.25	49.75
6[a]	42.5	2.5	—	—	2.5	2.5	—	—	—	0.25	49.75
7[a]	48	—	—	—	—	—	—	—	—	2	50
8[a]	48	—	—	—	—	—	—	—	—	2	50
9	38.4	—	9.6	—	—	—	—	—	—	2	50
10	28.8	—	19.2	—	—	—	—	—	—	2	50
11	19.2	—	28.8	—	—	—	—	—	—	2	50
12	9.6	—	38.4	—	—	—	—	—	—	2	50
13	4.8	—	43.2	—	—	—	—	—	—	2	50
14	—	—	—	48	—	—	—	—	—	2	50
15	—	—	—	—	—	—	48	—	—	2	50
16	—	—	—	—	—	—	60	—	—	2	38

[a] These preparations were varied by use of different grades of white soft paraffin (23).

conditions can then be deduced as in Fig. 2. The intersection of the Newtonian and non-Newtonian flow curves yields only an approximation of the rheological conditions during spreading. When the latter method is employed, the size of the region taken to represent the rheological conditions is arbitrary since there is only a single-point intersection. The size of the region (which may be affected by subjective factors) should be used to indicate the degree of error estimated to be involved in the determination and should be *at least* ± 2.5% of full-scale deflection. The magnitude of the error depends on such factors as the angle at which the Newtonian flow curve intersects the sample curve, the reproducibility of sample rheograms, and approximate allowances for slippage in the viscometer gap (particularly for some materials of high consistency).

Sensory Testing—The sensory testing of textural properties is widely used in the food industry (8–12) and, more recently, tests have been applied to cosmetic and pharmaceutical preparations for topical application (3–5, 13–15). Sherman (16, 17) recently reviewed textural profiling procedures with reference to both foods and topical applications.

The most commonly used scaling procedures (18) are:

1. Ordinal scaling—Two or more samples are required which contain the textural attribute being assessed at different levels. The samples are ranked (and scored) in an order believed to correspond with the level of the attribute. With an ordinal scale, the magnitudes of the differences between samples are not specified.
2. Interval scaling (Hedonic)—Two samples are used to represent the upper and lower limits of the level of an attribute on a linear scale. Other samples are allocated positions on the scale corresponding to the level of the attribute they possess.
3. Ratio scaling—One sample is taken as a standard and allocated an arbitrary number or score to define the level of a textural attribute it possesses. Other samples are then evaluated and allocated scores according to the ratio of the attribute levels possessed by each of them with respect to the standard sample.

Panel members may become fatigued if required to evaluate large numbers of samples in one test. For ordinal scaling procedures involving a large number of evenly distributed samples, the samples may be assessed in smaller groups and the scores from the individual groups may be unified. For example, if each of two small groups contains one of the same samples the ratio of the scores given to that sample in each test may be used to unify the two tests.

Other problems concerned with sensory testing are psychological. For example, sample sequence can affect results in food preference tests (19). Furthermore, it is difficult to determine exactly which parameter an individual is evaluating during a sensory test; the individual may evaluate the wrong parameter or may simply guess. The method of circular triads is a simple and effective check of a panel member's reliability (20–22). For example, consider four samples, 1, 2, 3, and 4, in which an attribute level is 1 > 2 > 3 > 4. Figure 4*a* is the triad system for these data completed by a consumer who is reliable. Figure 4*b* is the triad system for an unreliable consumer or panel member who found that 3 > 1 > 2 > 3 and that 4 > 3 > 1 > 4. This consumer is probably attempting to guess the results. The method may be expanded into a simple ordinal scaling procedure for any number of samples.

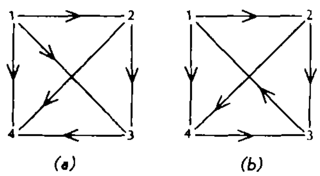

Figure 4—*The method of circular triads used in the assessment of the reliability of a consumer or panel member. Key: Consistent,* (a) *and inconsistent* (b) *triads, indicating that 3 > 1 > 2 > 3 and 4 > 3 > 1 > 4.*

EXPERIMENTAL

Preparations Used—Sixteen preparations were used with formulations as shown in Table I. All materials incorporated in the preparations were of BP or BPC quality, except sorbitan monooleate which was a commercial sample. The preparations were selected to cover the normal range of consistencies found in products designed to be rubbed into the skin.

Preliminary Study of Spreading Conditions—The arithmetic mean time of spreading of a panel of 10 persons spreading white soft paraffin onto the inside surface of the forearm was determined. The mean value was 19.3 ± 8.2 sec. The temperature of the forearm/fingertips was determined before and after spreading[1]. The arithmetic mean temperature was 33.9 ± 1.39°.

Instrumental Rheology—The viscosities of a range of Newtonian silicone oils were determined at 34 ± 0.2° using a Haake Rotovisko viscometer with the MV system of cylinders. This viscometer was employed to eliminate the risk of excessive slippage effects on the Ferranti Shirley viscometer due to a residual layer of silicone oil on the surfaces of the cone and plate. A Ferranti Shirley cone and plate viscometer with automatic flow curve recorder unit and X-Y plotter was employed in two modes to obtain rheograms for the test preparations at 34 ± 0.2°.

Instrumental Method 1—The samples were sheared from 0.0 to 1754.0 to 0.0 $sec.^{-1}$ in 120 sec. This produced a shear rate of approximately 580 $sec.^{-1}$ after 20 sec. When necessary, rates of shear greater than 2000 $sec.^{-1}$ were obtained by alteration (by a factor of 10) of the gear train in the cone drive-unit. Short sweep times are normally avoided, especially with the large cone, as derived data may contain instrumental artifacts due to inertial effects of the cone system. In the present work, it was necessary to use short sweep times to correlate the experimental time scale with the time of spreading determined previously. Inertial effects with the medium and small cones were negligible.

Instrumental Method 2—The Ferranti Shirley viscometer was used to determine shearing stresses at various constant rates of shear.

[1] With a thermistor (R.S. TH-B12) in conjunction with a Wayne Kerr Universal Autobalance.

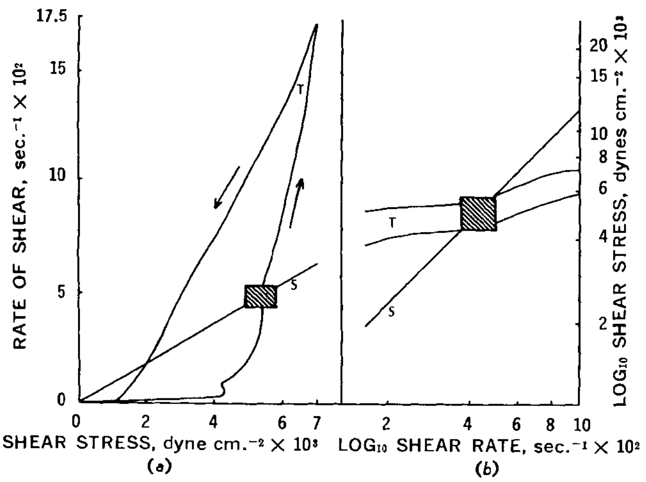

Figure 5—*Continuous* (a) *and discontinuous* (b) *shear flow curves (for Sample 4), indicating agreement between the two methods of obtaining the rheological conditions during spreading. S is a Newtonian silicone oil; T is Sample 4. Rectangles indicate approximate rheological conditions during spreading.*

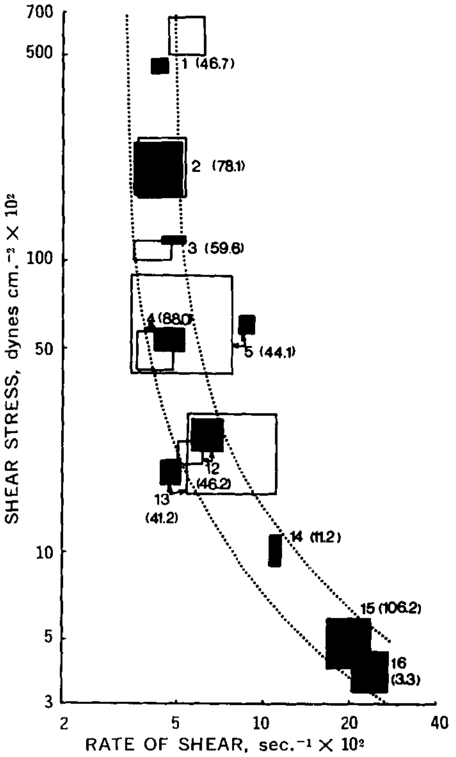

Figure 6—*Master curve of rheological conditions which occur during spreading of lipophilic materials on the skin. Shaded rectangles indicate data derived by Instrumental Method 1; open rectangles indicate data derived by Instrumental Method 2. The data are numbered to identify the samples; in parentheses are the coefficients of variation of the Newtonian viscosity values used to obtain the data.*

The "fast-up" control applied the rate of shear rapidly (within about 1 sec.) and the "hold-speed" control maintained the rate of shear. The shearing stress was determined initially and after 20 sec. (time of spreading) at each rate of shear.

Sensory Tests—A panel of 10 persons was used for all tests. The panel contained both sexes, with an age range from 20 to 50 years, and was not trained prior to the present work. The panel members were asked: (*a*) to ignore visual and initial sensations on the skin; (*b*) to ignore the absorption of a preparation by the skin, greasiness, tack, *etc.*; and (*c*) to assess the preparations according to the scaling procedure used, judged by the level of "stiffness, thinness, *etc.*," while spreading the preparations onto the inner surface of the forearm. In all sensory tests, samples were presented to panel members in random order and were maintained at 25 ± 1.0° prior to each test.

The reliability of the panel members was evaluated, using the method of circular triads with Samples 1–4, and was satisfactory.

Shear Conditions during Spreading—The panel members were given selected samples and asked to indicate for each the Newtonian silicone oil which appeared most similar while spreading on the skin. The panel members were also asked to spread a series of silicone oils onto the skin to enable an estimate of the velocity, V, of the fingers relative to the forearm to be obtained. To facilitate measurements, the subjects used linear strokes between marks 10 cm. apart on the forearm.

Ordinal Scaling—All 16 samples were used for the ordinal scaling test. These were presented to the panel in five groups, and the scores were unified in the manner previously described.

Ratio Scaling—The panel members were presented with Samples 4–13 inclusive. Sample 5 was chosen as a standard and given an arbitrary score of 5. The panel was asked to score the remaining samples on a scale from 0 to 10, judged by the level of spreadability of each sample relative to that of Sample 5.

Table II—Variation of Shear Rate, Velocity of Spreading, and Film Thickness with Apparent Viscosity of Preparation Being Applied

Silicone Oil, Newtonian Viscosity (poises) at 34°	Velocity of Spreading, cm. sec.$^{-1}$	Approximate Shear Rate[a], sec.$^{-1}$	Approximate Film Thickness, cm.
12.2	43	480	0.09
8.6	44	500	0.09
5.6	44	525	0.08
2.2	49	670	0.07
0.9	50	980	0.05

[a] Data obtained by superimposing Newtonian flow curves on Fig. 6.

Preference Testing—The panel was asked to evaluate Samples 4–13 inclusive using the following score system, which is of a type previously employed by other workers (2, 8):

Score	Sensation during Spreading
1	Too fluid, disagreeable
2	Fluid but all right
3	Agreeable
4	Stiff but all right
5	Too stiff, disagreeable

RESULTS

The rheological conditions operative during spreading of a topical preparation were determined. The shear stress/shear rate conditions were similar when determined by either adaptation of the Wood (8) method, *i.e.*, Instrumental Methods 1 and 2. A typical result is shown in Fig. 5. When the rheological conditions occurring during spreading of a range of products were used in a double logarithmic plot of shearing stress against rate of shear, a distinct trend was apparent, emphasized by the dotted lines in Fig. 6. Shaded areas on this master curve represent data derived using Instrumental Method 1; open areas represent data from Instrumental Method 2. The Newtonian viscosity used to determine the rheological conditions for any sample was the arithmetic mean of the silicone oil viscosity considered by each panel member to be similar to the topical product during spreading. The coefficients of variation of the data are indicated in Fig. 6.

The velocity of spreading of a series of Newtonian silicone oils on the skin was estimated. The approximate rate of shear during spreading of the oils on the skin was determined by plotting the Newtonian viscosities onto Fig. 6. The data were then used to calculate the approximate thickness of the film of a material formed during topical application using Eq. 1; results are given in Table II.

The data obtained using the three scaling procedures are given in Table III. The apparent viscosity values were derived from In-

Table III—Data Derived using Ordinal, Ratio, and Preference Scaling Procedures[a]

Sample Number	Apparent Viscosity, poises	Ordinal Scaling			Ratio Scaling			Preference Testing		
		S	*SD*	*CV*	*S*	*SD*	*CV*	*S*	*SD*	*CV*
1	96.2	14.4	1.1	8.0	—	—	—	—	—	—
2	44.0	13.6	1.1	8.5	—	—	—	—	—	—
3	27.0	8.7	0.9	10.3	—	—	—	—	—	—
8	14.2	8.0	0.7	8.4	7.4	1.2	16.6	4.4	0.7	15.9
9	11.3	6.0	2.1	34.5	5.0	0.0	0.0	4.2	0.6	15.1
5	11.0	6.8	2.9	43.7	7.2	2.1	29.2	4.3	0.8	19.1
4	10.8	4.9	0.6	12.2	—	—	—	—	—	—
10	10.6	4.1	0.5	11.3	4.6	1.2	25.3	2.9	0.3	10.9
7	9.4	6.6	0.9	14.1	5.6	1.9	33.9	3.5	0.5	15.1
11	6.3	4.0	2.0	50.0	2.6	0.9	36.5	2.6	0.8	32.4
6	5.3	4.0	1.8	45.5	2.9	1.4	48.3	3.2	0.4	13.2
12	4.2	2.6	2.2	83.5	2.0	1.1	53.4	2.0	0.8	40.8
13	3.9	2.4	2.1	87.4	2.1	1.0	47.4	1.8	0.6	35.1
14	1.0	1.1	0.3	26.5	—	—	—	—	—	—
15	0.3	0.9	0.2	24.7	—	—	—	—	—	—
16	0.2	0.8	0.4	52.7	—	—	—	—	—	—

[a] Samples arranged in order of apparent viscosity. *S* is the arithmetic mean of the data, *SD* is the standard deviation (determined using $n - 1$ degrees of freedom), and *CV* is the coefficient of variation expressed as a percentage.

strumental Method 1 data at the rate of shear indicated approximately by the master curve.

When the mean panel scores were graphically compared with their respective instrumental apparent viscosities, linear plots were obtained on semi and double logarithmic axes for the ratio and preference scaling procedure (Figs. 7 and 8, respectively). The ordinal scaling data were linear only on a double logarithmic plot (Fig. 9); there was an inflection in the curve at low values of apparent viscosity when plotted on semilogarithmic axes.

DISCUSSION

Figure 6 indicates that it is possible to derive the approximate rheological conditions which operate during spreading of a topical product upon the skin without previous knowledge of the film thickness, *d*, or the spreading velocity, *V*. Previous authors assumed that the rate of shear $\dot{\gamma}$, *V*, and *d* were effectively constant regardless of the consistency of the material (1–4, 6, 7). The data in Table II indicate that for Newtonian fluids, as the viscosity decreases, $\dot{\gamma}$ and *V* increase and *d* decreases. This *dynamic* relationship may explain former difficulties in evaluating both the rate of shear and the film thickness during spreading of topical preparations.

The master curve itself represents the approximate shearing stress/shear rate conditions which occur during spreading of products of the type used in this work on the skin. The curve may be utilized to determine the rheological conditions under which a product for topical application should be tested instrumentally for innovative work or for routine industrial control procedures. The sample is tested using Instrumental Method 2, which does not require an automatic viscometer, and the data are plotted onto the master curve (Fig. 10). The region of intersection approximately defines the rheological conditions which operate during spreading of the product and which should be simulated for instrumental control procedures. This method of determining the correct test conditions for a product has the advantage that the single master curve covers the range of lipophilic lotions, ointments, and pastes that are likely to be employed topically. Thus, Sample 1, zinc oxide and salicylic acid paste BP (Lassar's Paste) but with salicylic acid replaced by starch so as not to affect the skin during testing, was a stiff semisolid even at 34°, while Sample 16 was a mobile fluid.

The shape of the master curve is of interest; there are two main regions. The curve is approximately vertical at lower rates of shear, indicating that stiffer materials are assessed by a consumer at a relatively constant rate of shear. Shearing stress is presumably the variable assessed subjectively. Wood (8) found that the consistency of fluid foods was assessed in the mouth at a constant rate of shear of approximately 50 sec.$^{-1}$, with shearing stress as a variable. However, the spreading of low viscosity materials on the skin occurs at increasing rates of shear and decreasing shearing stresses as the product consistency decreases. Possibly the stimulus assessed here is the ratio of the two variables. It is thus possible that the consumer uses two operative regimes when judging the spreadability of topical preparations on the skin. There would, of course, be considerable overlapping at intermediate consistencies. However, the subjective evaluation of textural properties may not be so simple. Scott Blair (24) suggested that certain textural properties were amenable to dimensional analysis and could be represented by a power equation of the type proposed by Nutting (25). For example, the consistency,

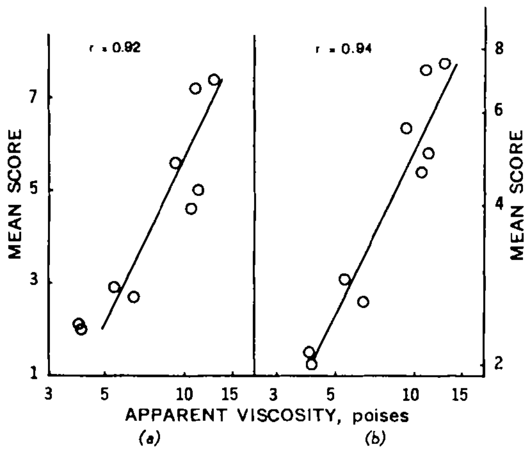

Figure 7—*Semilogarithmic* (a) *and double logarithmic* (b) *plots of ratio scaling data against apparent viscosity; r is the correlation coefficient.*

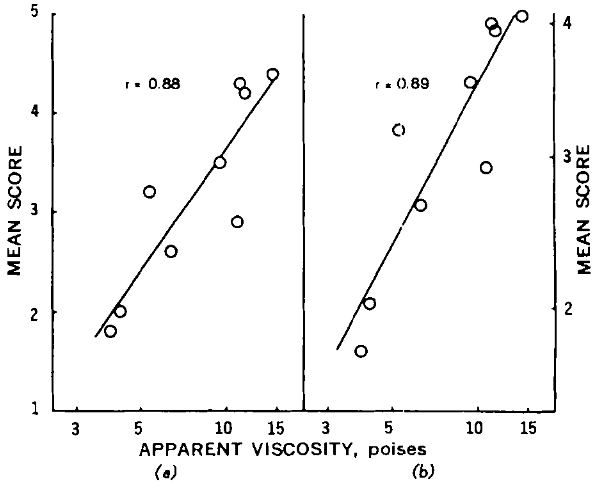

Figure 8—*Semilogarithmic* (a) *and double logarithmic* (b) *plots of preference test data against apparent viscosity; r is the correlation coefficient.*

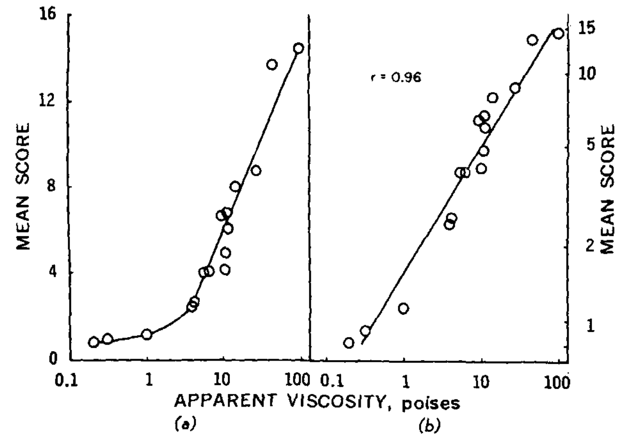

Figure 9—*Semilogarithmic* (a) *and double logarithmic* (b) *plots of ordinal scaling data against apparent viscosity; r is the correlation coefficient.*

or a related attribute, may be defined at constant stress by the equation:

$$\theta = \sigma\gamma^{-1}t^{a} \qquad \text{(Eq. 2)}$$

where θ is the textural attribute; σ is the stress; γ is the strain; t is the time; and the dissipation coefficient, a, has a value of 1 for viscous fluids and of 0 for elastic solids and is fractional for viscoelastic materials.

The three scaling tests performed by the panel clearly indicated the ability of the panel to distinguish between the spreading properties of the preparations used. By using Instrumental Methods 1 and 2, it was found that slippage effects occurred significantly at high rates of shear with the stiffer preparations. The phenomena of sample fracture and slippage were discussed previously (5). However, it was found that the instrumental data correlated with the sensory data; it is, therefore, possible that sample fracture and subsequent slippage may also occur on the skin. Slippage effects using cone and plate viscometry were discussed by Boylan (26), who also investigated the rheological conditions associated with topical application. However, the maximum rate of shear employed by Boylan (26) was 270 sec.$^{-1}$, a value he obtained from the literature, and thus the work is not directly comparable with the present study.

Plots of the ordinal scaling data against apparent viscosity produced a curve with an inflection when plotted on semilogarithmic axes and a linear graph when plotted on double logarithmic axes (Fig. 9). The inflection in the semilogarithmic plot may correlate with the general shape of the master curve and support the proposition that the consumer applies two separate mechanisms or regimes for sensory evaluation of spreading of topical preparations, depending on the overall consistency of the product. As stated previously, ordinal scaling does not indicate the magnitude of the differences between samples; it merely indicates whether the panel members are able to distinguish the differences. In this test the differences in the levels of spreadability of the preparations were distinguished by the panel members. However, this test and other ordinal tests are of limited use, because the shape of the curve obtained depends on the spacing of samples based on their attribute level. Relatively even spacing yields linear graphs; randomly grouped spacing would not necessarily do so.

Plotting of the ratio scaling data *versus* apparent viscosity produced linear graphs on semi and double logarithmic axes (Fig. 7). This indicated that the correlation between stimulus and perception may be represented by logarithmic or double logarithmic (power) equations as proposed by Weber and Fechner (27) and Stevens (28), respectively. The equations are:

$$\theta = K \log S \qquad \text{(Eq. 3)}$$

$$\theta = K_1 S^{n} \qquad \text{(Eq. 4)}$$

or:

$$\log \theta = K_2 n \log S \qquad \text{(Eq. 4a)}$$

where θ is the sensory response to a stimulus, S; and K, K_1, K_2, and n are constants. In these tests, θ corresponds to panel score and S to apparent viscosity (or shear stress). The arbitrary score given to the standard preparation in a ratio scaling procedure affects the value of the constant, n; standardization of the scoring procedure may indicate that the value of n has some significance with regard to the type of stimulus.

While the correlation coefficients for the regression lines in Fig. 7 were high, they should not be relied upon for predictive inferences (29). The correlation coefficients indicate the tendency of the spreadability and the apparent viscosity to vary in a consistent manner over the range of conditions studied. The fact of correlation does not necessarily imply anything about cause and effect. The observed relationship may not remain consistent over a wider range, especially in extreme conditions.

The regression lines obtained using the preference test data (Fig. 8) may also be represented by double or single logarithmic equations. Stevens (28) defined two types of stimulus: prothetic (quantitative, how much?) and metathetic (qualitative, of what kind?). The former are represented by double logarithmic equations (Eq. 4) and the latter by single logarithmic equations (Eq. 3). The ratio scaling test may be classified as prothetic in nature, because the panel assessed *how much* more or less stiff each sample was compared with a standard sample. The preference test was metathetic since the panel evaluated the *type* of sensation, *i.e.*, agreeable or not agreeable. The data for the two types of test should, therefore, be represented by Eqs. 3 and 4 for preference and ratio procedures, respectively. Because the two sets of data may be represented adequately by either type of equation (as shown in Figs. 7 and 8), the classification suggested by Stevens (28) may not be valid in this case. Langenbucher and Lange (2) found single logarithmic relationships between panel preference scores and Newtonian viscosity for extrusion and topical application tests.

The optimum apparent viscosity for spreading, determined from Fig. 8 for a score of 3, was approximately 6.8 poises. The range of acceptable apparent viscosity, lying between scores 2 and 4, was 3.9–11.8 poises. By plotting out these values of Newtonian viscosity onto the master curve, it is possible to define a *preferred region;* this indicates the shear stress/shear rate conditions acceptable to the average consumer. The bounds of the region (indicated by the shaded rectangle in Fig. 10) are approximately 400–700 sec.$^{-1}$ and 2000–7000 dynes cm.$^{-2}$.

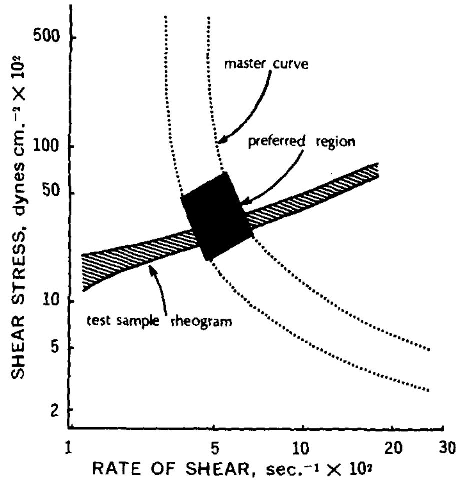

Figure 10—*Spreadability master curve with superimposed preferred region and test sample rheogram.*

The work discussed here indicates the type of laboratory screening procedure which may be utilized in innovative work prior to field trials. The new product(s) is assessed using Instrumental Method 2 to obtain a rheogram. When the instrumental data are plotted onto the master curve (Fig. 10), the two curves should intersect within the preferred region and near to the position indictated by the optimum spreading viscosity. Such a product would have a spreadability with maximum consumer acceptance potential.

The rheological conditions indicated by the preferred region hold only for materials of the type used in this work. The location and shape of a master curve for other types of materials, such as gels, foams, and aqueous emulsions, may be different. For example, Langenbucher and Lange (2) found that the optimum viscosity for the spreading of Newtonian silicone oils on the skin was 1.0 poise; the acceptable range was 0.2–5.0 poises at 30–35°. The rate of shear estimated was 10^4 sec.$^{-1}$.

The three scaling procedures employed in this work appear to be of similar efficiency in differentiating and ranking the preparations used, since the correlation coefficients of the regression lines of the double logarithmic plots are similar. Linear graphs may also be obtained by plotting the score data against shearing stress (determined at the shear rate indicated by the master curve) rather than the apparent viscosity. Preference testing has the obvious advantages that it may be used to determine the textural conditions which will achieve optimum consumer acceptance and it may be used in the design of laboratory screening procedures for new products.

The data reported here may tend to indicate that individual panel members, and hence the consuming public, behave similarly when evaluating spreadability. The linearity of the data when plotted in Figs. 7–9 especially gives this impression. However, all the data employed in these diagrams are arithmetic mean values. The assessments made by individual panel members varied considerably. The coefficients of variation (ratio of standard deviation to the mean, expressed as a percentage) of the individual data points (Fig. 6 and Table II) indicate the wide spectrum of sensation produced by the same stimulus upon different panel members. Causes of the variation may be: (*a*) lack of training of panel members in this type of work; (*b*) variation in skin temperature, which will affect the rheological properties of the samples and thus their spreadability; (*c*) variation in the thickness of the film being spread, which greatly affects the shear rate achieved; and (*d*) general biological variability.

SUMMARY

The work showed that it is possible to derive a master curve of the rheological conditions which operate when lipophilic lotions, semisolid emulsions, and pastes are spread on the skin. The master curve may be employed in the design of suitable instrumental tests for industrial product control procedures concerned with spreadability.

The methods used to derive the spreadability master curve may be applied to other systems and other textural parameters. With water-miscible creams, for example, the film thickness, *d*, during spreading may be lower than that found in this work, and the shear rates involved in spreading will thus be higher. Other textural properties for which master curves may be constructed are the initial consistency of a product and visual evaluations of viscosity, which apply to semisolids and fluids, respectively. The techniques are not limited to the pharmaceutical industry. Cosmetics are applied to the skin in the form of lotions, creams, ointments, and materials of even greater consistency such as lipsticks; sensory evaluations are more important in the cosmetic field than in pharmacy. The food industry may also benefit from the types of procedures outlined here. A range of textural properties, concerned with viscosity assessment in foodstuffs, could be more easily dealt with using the master curve method.

Ordinal, ratio, and preference scaling procedures were found to be of similar efficiency in detecting the ability of the panel to differentiate levels of spreadability in the preparations used. The master curve and preference test data were used to indicate the type of spreadability screening test that may be employed in innovative work prior to more extensive field trials. The application of such a test may lead to savings in time and money.

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