

Four methods for the characterization of dentifrices and other semisolids

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Synopsis—During the course of development work, the need arose for characterization and comparison of batches of DENTIFRICES. Four methods used in other technologies are reported for their utility in the evaluation of these and other SEMISOLIDS.

Force-time traces were recorded on a modified *Instron* TENSIO METER for a piston moving at constant speed down a full tube of toothpaste and again down the emptied one. The difference was the corrected initial FORCE for EXTRUSION. At the same time, a number of extruded drops were weighed. The mean weight of a drop was divided by the diameter of the orifice to obtain the TENSILE STRENGTH of the paste.

A polyethylene disc resting on the surface of a jar of dentifrice was withdrawn at constant speed on an *Instron* tensiometer. The force-time curve showed a characteristic maximum cohesive force. The final value obtained on separation of paste and disc was a measure of STRINGINESS.

A notched doctor blade was used to apply 10 parallel stripes of uniform width but increasing height upon a surface. Rotation through 90° permitted sag of the semisolid, giving a stripe number characteristic of RESISTANCE to SAG.

INTRODUCTION

Numerous semisolids are in household use to-day, ranging from medicinal ointments, skin creams and dentifrices in the bathroom, to sandwich spreads, mayonnaise, whipped cream and jellies in the kitchen,

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and to putty, polishes, grease and paint in the workroom. Thermoplastics, ceramics, glass and other materials appear rigid at room temperature but are processed in the semisolid state. The measurement and control of rheological properties of such materials presents great difficulties. Historically, each craft and later each industry has tried to solve its particular rheological problems in isolation, so that to-day a vast amount of knowledge is available but dispersed among numerous specialized sectors of technology.

During the course of product development on dentifrices, the need arose for characterization and comparison of batches of pastes. Four methods were transferred from other technologies and are reported for their potential utility in the evaluation of semisolids. The first two methods utilize the principle of extrusion, the next measures values related to cohesion and adhesion and the fourth ascribes a number to the property of sag.

EARLIER WORK ON EXTRUSION

A variety of simple rheometers has been used to measure the extrusion of butter (1), fats (2-4), molten lead and wax (5), clay pastes (6), mastics (7) and foods (8). Prentice (9, 10) and later Wiedermann (11) found a good correlation between the ease of extrusion of fats and their spreadability. Further measurements on the extrusion of foods through simple devices have been made (12-16). The force for extrusion of ointments and pastes from tubes and bottles has also been measured (17-20). The ductility of petrolatum has been measured by extruding a column vertically downwards, permitting it to break under its own weight and measuring the length of the residual cone adhering to the orifice (21). Food texture has been assessed by back extrusion through the concentric annulus between a cup and a metal plunger (22-24).

EXPERIMENTAL

Materials

A number of viscous liquids, suspensions, gels and pastes were prepared for this study.

Most mucilages consisted of a dispersion in 70% sorbitol syrup of gums and other hydrocolloids, such as Carbopol 940, a carboxy-vinyl polymer

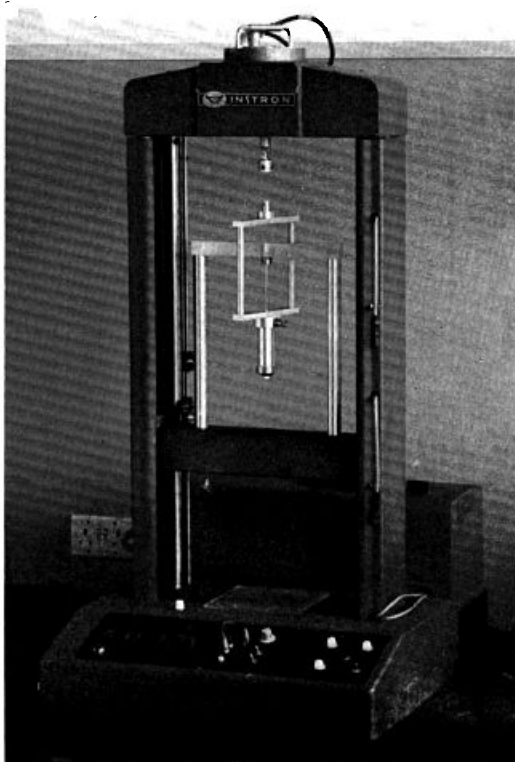


Figure 1. Instron tensiometer with Mk I extrusion rheometer.

which was neutralized with sodium hydroxide. Another consisted of a 20% suspension of a synthetic silica thickener in sorbitol.

Dentifrice 1 contained 14% of an abrasive, dentifrice 3 contained 32% of a mixture of abrasives, Seven different hydrocolloids were used at the stated concentrations, with corresponding minor changes in water content, in order to achieve acceptable consistency and different rheological properties (*Table I*).

Methods of rheological characterization

Initial force of extrusion (corrected)

Vasic and deMan's method (16) was adapted for use on an *Instron* tensiometer.

Extrusion rheometer attachment. Figs. 1-3 illustrate the apparatus, which consisted of a motor crosshead rigidly joined to the *Instron* crosshead. A stainless steel rod fitted to an aluminium piston was attached to the motor crosshead. The piston was fitted at first to a metal tube bearing a brass cap,

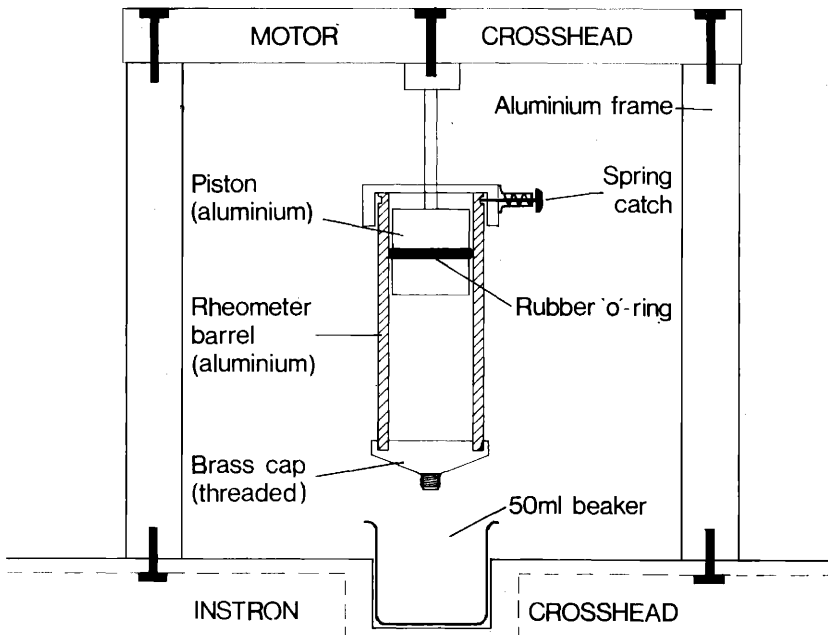


Figure 2. Mk I rheometer barrel.

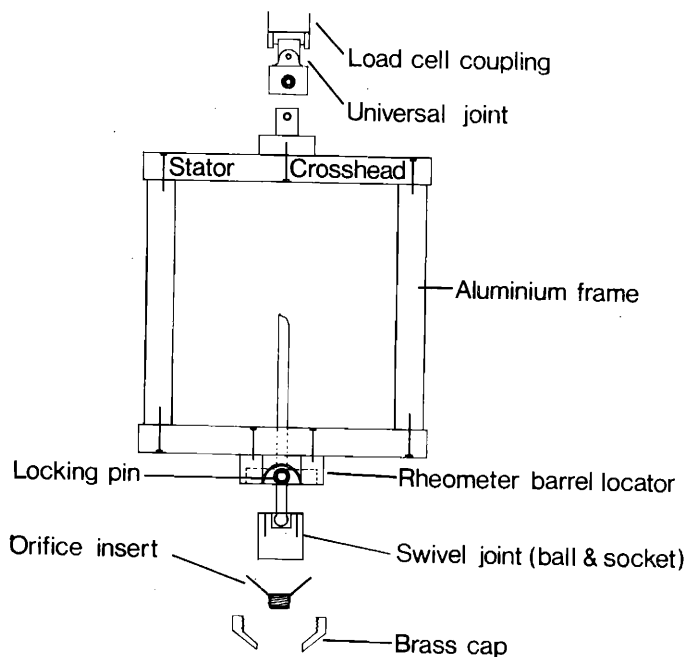


Figure 3. Instron crosshead assembly.

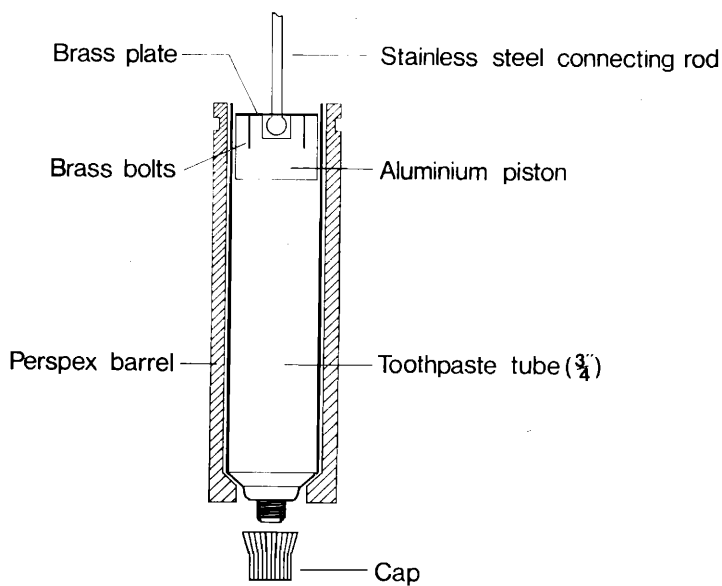


Figure 4. Mk II rheometer barrel.

which held the orifice insert, *Fig. 2*. It was later found more convenient to fit the piston to a standard, inverted, uncrimped tube of dentifrice supported in a *Perspex* holder, *Fig. 4*. The latter was attached to the *Instron* crosshead, which was linked as usual by a pin to coupling of the load cell.

Experimental procedure. Tests with a stopwatch and dentifrices extruded manually from standard tubes showed an extrusion velocity of about 1.3 cm sec^{-1} . Speeds of 5 and 10 cm sec^{-1} for the *Instron* crosshead corresponded to extrusion velocities of 0.9 and 1.8 cm sec^{-1} from the tube. Inverted tubes were filled to a fixed height, stoppered and left for 24 h. They were then placed, one at a time, in the *Perspex* barrel. The screw cap and rubber stopper were removed and the piston was inserted in the inverted tube. As the crosshead began to descend and the piston began to extrude the paste, the force for extrusion, which was recorded, rose sharply; the rapidly attained maximum was taken as the 'initial force for extrusion'. A repeat run was made with the empty tube. The initial force for extrusion recorded on the emptied tube, due to friction between piston and the aluminium walls, was subtracted from the initial force for extrusion for the filled tube.

Results. *Table I* shows the results for the initial force for extrusion determined at two shear rates, corresponding to consumer practice when extruding dentifrice from this standard size of tube. *Table II* shows the effects of time of storage on this parameter.

For most pastes, there was an increase in the force required for extrusion with increase in velocity of extrusion, as was expected. *Table II* shows that for most pastes, the initial force required increased with the time elapsed between filling of a tube and extrusion.

The modified cellulose thickener in dentifrice 1 was the only material to show a decrease in initial force for extrusion with increasing speed. The two types of xanthan gum showed a remarkably low force for extrusion in both dentifrices, as can be seen in *Table II*. The effect persisted in a mixture with sodium carboxymethyl cellulose.

A small panel of laboratory staff confirmed that ease of extrudability of the dentifrices tested varied directly with the initial force for extrusion.

Discussion. The initial force for extrusion from a new tube of dentifrice is particularly important for the consumer, who makes a judgement on the quality of the product from this sensory cue. With some pastes, the force required varies dramatically with temperature and this could prove unsatisfactory in either summer or winter. There seems to be a range of acceptable pressures for deforming the tube and extruding the paste, outside which products are judged unfavourably. Although it is possible to alter

Table I. Initial force for extrusion (f) and tensile strength (t) at two shear rates

Material	Thickener	Force (f)						Tensile strength (t)					
		5 cm sec ⁻¹		10 cm sec ⁻¹		5 cm sec ⁻¹		10 cm sec ⁻¹		5 cm sec ⁻¹		10 cm sec ⁻¹	
		f	Range	f	Range	t	Range	t	Range	t	Range	t	
Dentifrice 1	0.3% modified cellulose	340	320-360	300	250-350	5.9	5.8-6.0	5.2					
	0.3% SMC A	520	490-540	570		5.3	5.2-5.4	5.7					
	0.6% carrageenan A	680	550-720			18.5	18.0-19.0						
Dentifrice 2	0.8% carboxy vinyl polymer	150	100-250	170		3.5	3.0-3.7	4.4					
	0.75% SMC B	775	740-800	1050	1000-1085	7.5	7.4-7.6	9.0					
	0.75% xanthan gum A	370	330-420	480	470-500	4.6	4.4-4.7	5.3					
70% sorbitol	20% silica gel	640	570-700	900	600-1150	1.7	1.5-1.9	2.4					
	0.5% carboxy vinyl polymer	360	300-400	560		7.0	6.9-7.1	7.4					

Table II. Initial force for extrusion and tensile strength after 1 and 2 days

Material	Thickener	Initial force		Tensile strength	
		1 day	2 days	1 day	2 days
Dentifrice 2	1.2% modified cellulose	600, 400	780, 750	9.6, 10.5	11, 18
	0.9% SCMC B	670, 760	960, 1160	5.5, 5.7	
	1.0% xanthan gum B	105	295, 260, 260, 260	2.7	2.7, 2.8
	0.5% SCMC B with 0.3% xanthan gum A	440, 460	530, 590, 620	4.2, 4.2	
	1.1% carrageenan B	390, 435	620, 620	4.2, 4.2	
	1.3% carrageenan A	370, 370	450, 495	3.4, 3.5	
Dentifrice 3	1.5% SCMC B	650, 685	780, 820	6.6, 6.3	9.0, 10.3, 9.6
	1.7% xanthan gum B	190, 170		4.9, 4.9	

the pressure required by changing the diameter of nozzle on the tube, a change in formulation is generally more convenient. Of course, when different batches of toothpaste are to be compared, the force required to deform the tube need not be taken into account.

In his work on the horizontal extrusion rheometer, Prentice (8) considered the force of extrusion to consist of two parts (*Fig. 5*). The force required to push the sample of cooking fat along the cylinder depended upon the friction between metal and fat and was found to decrease linearly with length of stroke. The force required to drive the sample through the orifice was considered constant and was of major interest to this investigator.

Although Prentice (9, 10) and Hoffer (25) found a linear decrease in force with time, that is, decreasing length of stroke, as cooking fats, margarine and butter were extruded through the orifice, this was not found to be the case with dentifrices on a *FIRA-NIRD* extruder* (26). The smooth brass tubes had been filled with pastes and extruded after 24 h. A most irregular series of variations of force with time was obtained. These variations were attributed to the friction of hard particles of abrasive wedged between piston and tube.

Vasic and deMan (16) were interested in the force required to push material along the tube as well as that required for extrusion through the orifice and used the average force on one downstroke in their calculations. They repeated the run with the emptied cylinder and subtracted this force from the average.

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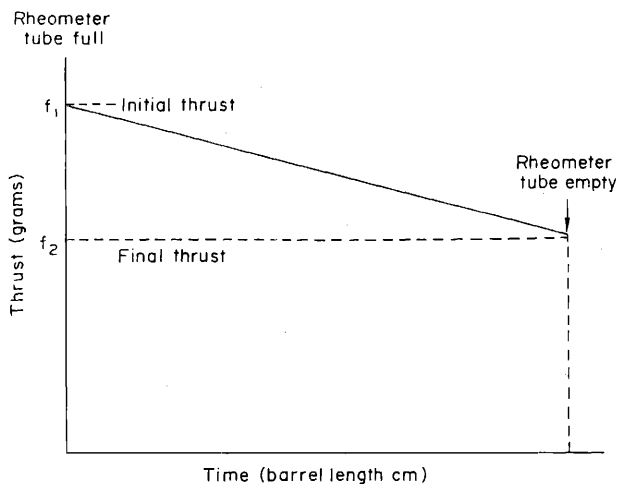


Figure 5. Force for extrusion—idealized.

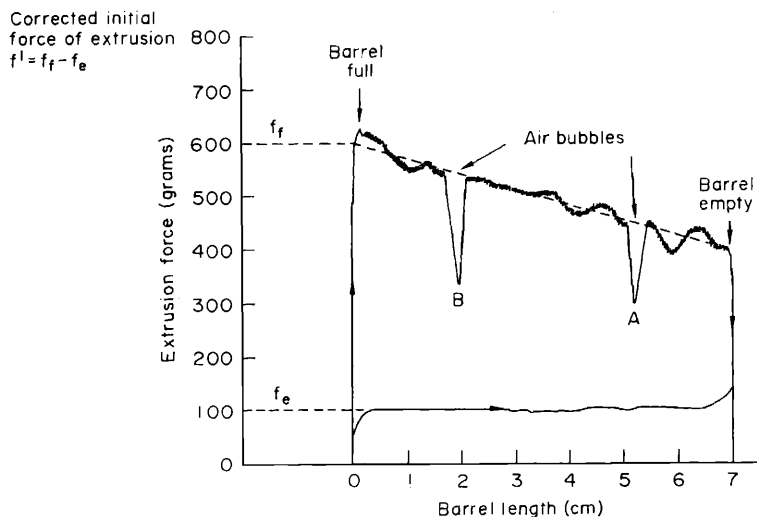


Figure 6. Force for extrusion—actual. Crosshead speed = 5 cm min^{-1} .

In this work the maximum, initial force required for extrusion was considered of greatest interest. Considerable variations were found on samples from the same batch stored for the same length of time in separate tubes. These variations were considerably reduced when the initial force for extrusion for each emptied tube was subtracted from the initial force determined on the full tube, Fig. 6.

Tables I and II show that despite the scatter of experimental results, the method can usefully detect differences in formulation between similar toothpastes.

Characterization by tensile strength

Ben-Arie (27) pointed out that much apparatus for the measurement of rheological properties of viscoelastic materials, such as 'gel strength', was adapted to specific uses and that the experimental values were dependent upon the specific equipment employed. Some devices measured rigidity, some plasticity and others elasticity. Moreover, there was generally no physical correlation between the results of the different methods. On the other hand, tensile strength is a clear, well-defined concept.

Ben-Arie (27) extruded Napalm gels with piston and barrel downwards through various nozzles. A number of drops extruded at constant speed were weighed. The mean weight of a drop divided by the diameter of the nozzle was the tensile strength. He established that the experimentally determined values were independent of the areas and of the area-length ratios of the nozzles used for extrusion. As the tensile strength increased linearly with velocity of extrusion, the values were determined at two convenient velocities and extrapolated to zero.

Charm (28) extruded mayonnaise and ketchup through tubes using air pressure and calculated the tensile strength in a similar way. The diameter of the tubes had to be below that critical value at which the material would flow under gravity.

For this work the tensile strength at the velocity of extrusion used for dentifrices was of interest, and Ben-Arie's method was employed.

Experimental procedure. The extrusion rheometer was used to determine the initial force for extrusion and the tensile strength of the paste in one run. The drops and pieces of extruded ribbon falling from the rheometer after extrusion were simply counted and weighed, *Fig. 7*. The mean weight of a drop was divided by the diameter of the nozzle to obtain the tensile strength.

Results. *Table I* shows the results for two velocities of extrusion and *Table II* the effect of increasing times of storage on a number of formulations. For most pastes there was an increase in tensile strength with increased velocity of extrusion (or driving pressure) and with increasing time of storage. Only the modified cellulose thickener in dentifrice 1 showed a decrease in tensile strength with increasing velocity of extrusion. The xanthan gums in dentifrice 2 showed a remarkably low tensile strength both 1 and 2 days after filling.

A small panel confirmed that ease of extrudability of the dentifrices tested varied inversely with the tensile strength.

Discussion. Determination of tensile strength was found to be simpler and more reproducible than determination of initial force for extrusion. The necessary equipment can be much less sophisticated than the *Instron* tensiometer and of course no recording equipment is required.

Many dentifrices increase in tensile strength with time of storage. Indeed, a low degree of thickening immediately after manufacture, permitting easy filling of tubes, followed by development of structure over a period of several weeks, has been claimed as an advantage for one type of thickener (29). In fact, shear-thinning is highly desirable in dentifrices; the paste should be easy to squeeze out of its tube to form a firm ribbon on the brush. In the mouth it should then again rapidly deform under shear. With most toothpastes, this is a thixotropic, time-dependent process. Measurement of tensile strength at two time intervals after filling, as in *Table II*, would distinguish between the slow time-dependent and other processes.

The speed of the method and the low loss of heat to the surroundings due to the *Perspex* support permit measurements to be made over a range of temperatures.

Characterization by maximum cohesive force and stringiness

Claassens (30) developed the so-called 'hesion' balance for measuring the adhesion-cohesion of butter to various materials. The vertical pull required to separate butter from a solid disc was determined. Henry and Katz (31) adapted this method for use with the *Instron* tensiometer and measured the adhesion and stringiness of starches, gums and whipped cream to solid surfaces. Their method was adopted for use on gels and dentifrices.

Experimental procedure. A polyethylene disc of 2.5 cm diameter was fixed to a spindle attached to the jaws of the calibrated *Instron* tensiometer and a jar containing the paste was raised until complete contact was made all around the disc, care being taken to avoid entrapment of air between disc and gel. A time for equilibration was allowed, according to the degree of thixotropy of the semisolid. The crosshead was then started and a force-time curve was recorded, *Figs. 8-10*. The stringiness or S-factor was measured just after the thin neck of semisolid had ruptured.

Theory. As the disc was being lifted at constant speed, the abscissa in *Fig. 10* has been plotted as distance in cm rather than time in seconds. The force-distance curve is in many ways similar to those for metals with respect to three well-defined regions:

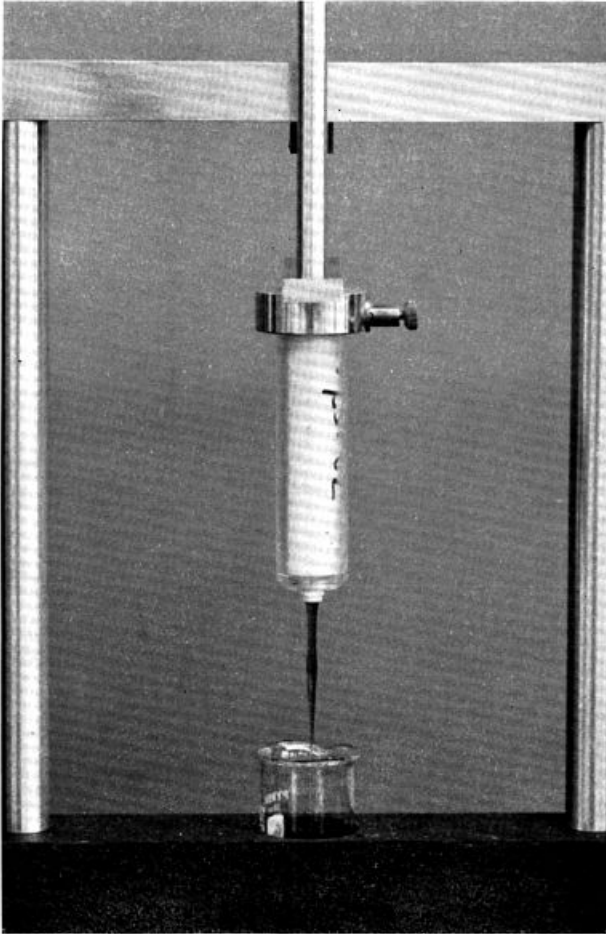


Figure 7. Mk II rheometer with toothpaste tube insert.

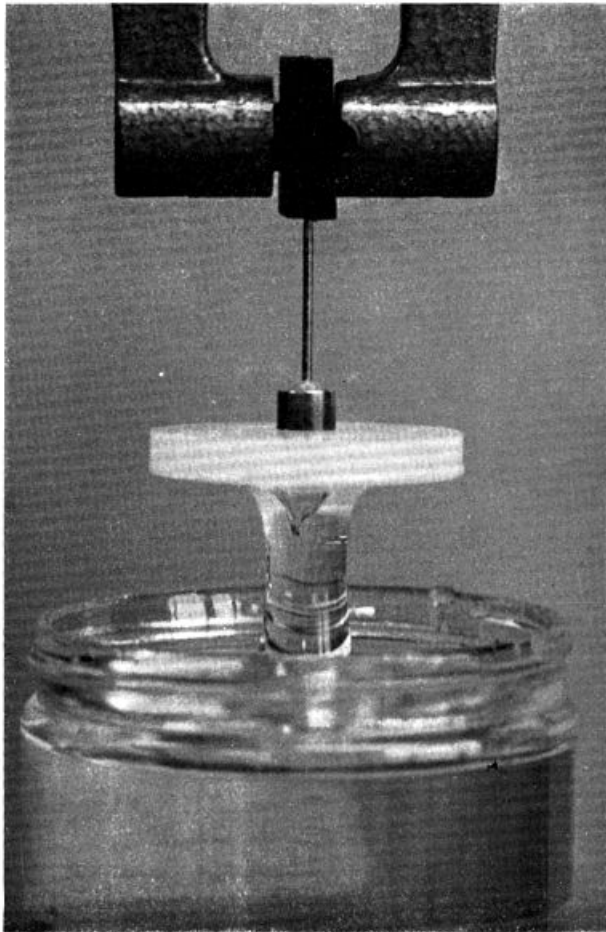


Figure 8. Hesion apparatus—'Necking' of 2% SMC gum in 70% sorbitol after extension for 50 sec.

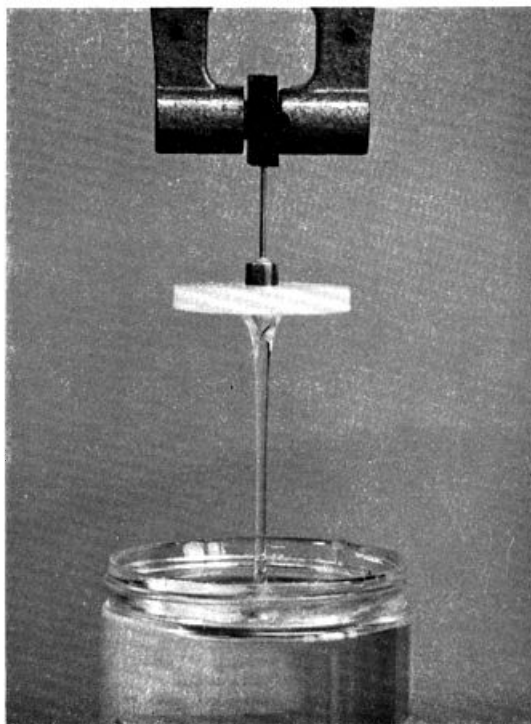


Figure 9. As in *Fig. 8* after 100 sec.

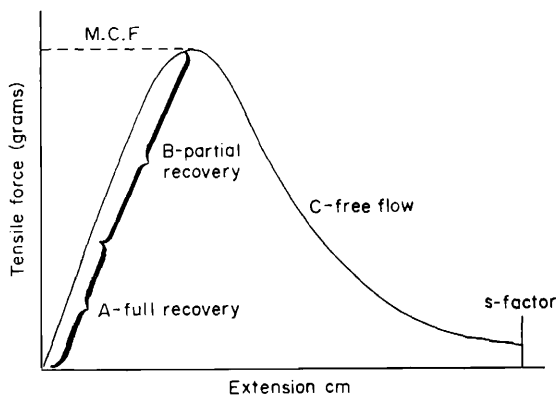


Figure 10. Force-distance curve during adhesion-cohesion.

- A — in this elastic region total recovery occurs after deformation;
- B — recovery occurs with some deformation;
- C — beyond the point of maximum cohesive force, recovery cannot take place and so free flow occurs until the point of rupture, characterized as the stringiness or S-factor.

Henry and Katz (31) pointed out that the area under the force-extension curve was proportional to the work done in extension. The total force reached a maximum value and diminished before the extended semisolid ruptured. Although the tensile force per unit area did not decline, the total force ($\text{g cm}^{-2} \times \text{area in contact}$) reached a characteristic maximum value and diminished before rupture.

Results. Table III shows the maximum cohesive force and the stringiness for a number of pastes and mucilages at two crosshead speeds. While the maximum cohesive force increased or remained constant with increasing speed, the S-factor increased in all cases. For all systems studied, the maximum force was reached fairly quickly, indicating that this was the force required to lift the disc over the first few millimetres from the surface. It was found to be dependent upon the time required to develop thixotropy, as shown by successive trials with material from the same jar. Figs. 11 and 12 show the effects of differing crosshead speeds on the shape of the force-extension curves for two materials. The maximum cohesive force increased with increasing crosshead speed for the mucilage but decreased for the dentifrice.

Discussion. The maximum cohesive force and stringiness were found to be useful parameters for characterizing dentifrices and mucilages, especially

Table III. Maximum cohesive force (MCF) and stringiness at two crosshead speeds

Material	Thickener	MCF		S-factor	
		5 cm min ⁻¹	10 cm min ⁻¹	5 cm min ⁻¹	10 cm min ⁻¹
Dentifrice 1	0.3% modified cellulose	240	—	1.2	—
	0.8% Carboxy-vinyl polymer	56	—	3.5	4.4
	1.2% Alginate A	215	295	5.6	7.9
	1.0% Alginate B	80	—	9.9	—
	70% Sorbitol	0.5% Carboxy-vinyl polymer	150	185	3.3
	1.5% Alginate C	70	95	> 16	> 16
	1.5% Alginate D	38	45	> 16	> 16
	1.5% Alginate E	40	40	12	> 16
	0.9% Carragheenan A	64	70	2.4	7.0
	0.7% Carragheenan A	58	65	2.8	3.5
	2.0% SCMC C	54	68	> 16	14.2
	1.7% SCMC C	20	25	9.2	15.0
	20% silica	44	—	8.8	11.5
	19% silica	24	—	1.3	—
	1.2% SCMC B	—	—	7.5	—

when used in conjunction. Acceptable dentifrices had S-factors below 4 cm at a rate of extension of 5 cm sec⁻¹.

For quality control of pastes and gels a far simpler instrument could be cheaply constructed.

Characterization by measurement of sag

It is a common experience that after extrusion from a tube upon a toothbrush, some dentifrices tend to sag or slump between the bristles. With pastes of low dispersibility, sag would increase the time required to clean the toothbrush after use. The slow ooze of toothpaste from an open tube left in the bathroom is probably also associated with the property of sag. From these considerations, it seems likely that the overall preference for a toothpaste may suffer if the property of sag falls below a certain limit.

The tendency to sag is a characteristic property of a semisolid and may be measured simply without reference to a container or to a particular procedure for extrusion. This has been done in the paint industry. In the paint laboratory, many different coatings, which may vary in their requirements as regards resistance to sag, are being tested and the chemist requires a simple, rapid and reproducible method for specifying and testing resistance to sag. The method should have good precision, with test results preferably expressed in unequivocal numbers (32).

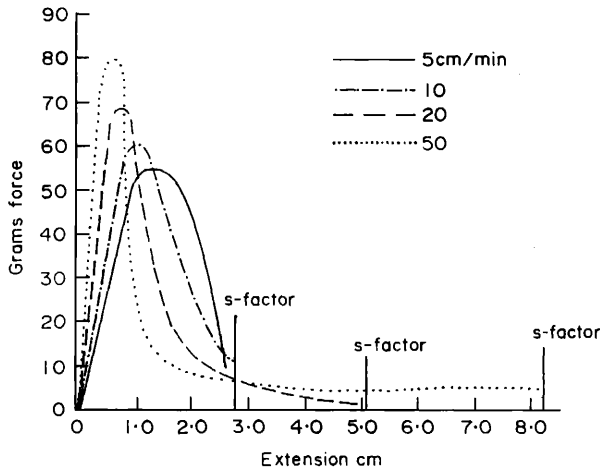


Figure 11. Force-distance curve for carragheenan A in sorbitol syrup.

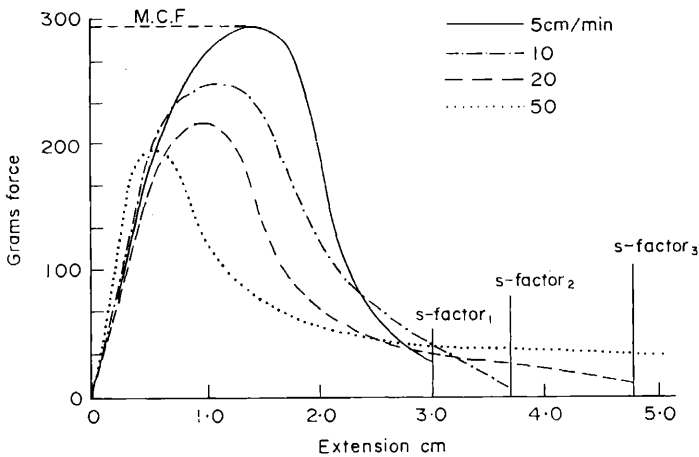


Figure 12. Force-distance curve for a dentifrice.

Reynolds and Larsen (33) devised a sag index blade for the purpose and this was improved by Schaeffer (32). It consists of a doctor blade with a series of wide notches of varying clearance which is used to lay down a series of parallel stripes of paint of uniform width but increasing height. Sagging is related to the height of the stripe. The blade is commercially* available in three sizes: 1-6 mils for spraying and dipping enamels, 3-12 mils for trade sale type paints and 14-60 mils for coatings of high film thickness

* The Leneta Company, Ho-Ho-Kus, New Jersey, U.S.A.

(1 mil = 10^{-3} inch). Its use is mandatory for measuring resistance to sag in some US Federal Specifications.

Description and procedure. For use on dentifrices, a simplified sag meter in the range 60–120 mils was constructed (*Fig. 13*). The brass doctor blade has 11 notches, each $\frac{1}{4}$ in. wide with intervening gaps of $\frac{1}{16}$ in. The heights of the notches are 60, 70, 80, 90, 100, 120, 140, 160, 180 and 200 mils. The last notch is a thin, shallow marker.

Toothpaste is placed upon a horizontal, glazed glass plate along the recessed side of the meter, which is drawn at a slow, steady rate along a straight edge. A series of equidistant, parallel stripes of uniform width and varying heights is obtained. The plate is placed in a vertical position with the stripes horizontal, the fattest stripes being lowest.

A slightly roughened surface is required to prevent slip of the stripes of toothpaste.

Results. *Fig. 14* shows the results of a typical sag test. The five fattest stripes have sagged, obscuring the intervening gaps. (The lowest stripe is the shallow marker.) The sag number is therefore 5. A series of pastes can be compared easily in this way, sag ceasing after about 30 min.

For a series of more fluid pastes, the time taken for the most mobile one to show a sag number of 1 may be noted. The other pastes can then be ranked in order of sag number, using the same time span. *Fig. 15* illustrates the good reproducibility of the test. Each horizontal line shows the

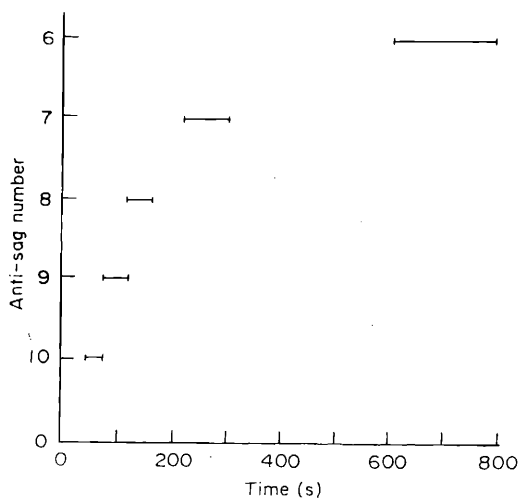


Figure 15. Span results for five trials on one paste. Final sag number for all trials 5.

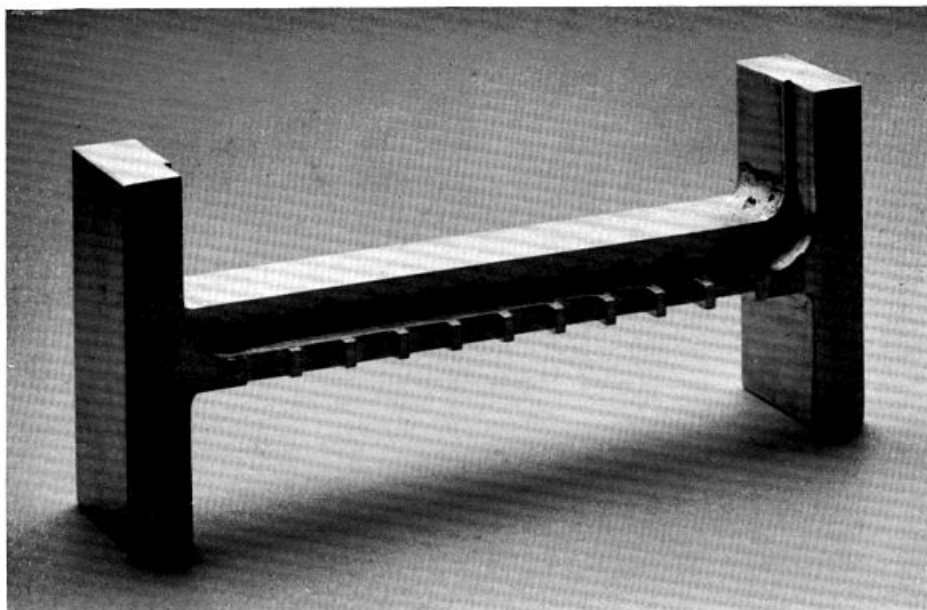


Figure 13. The toothpaste sag meter.

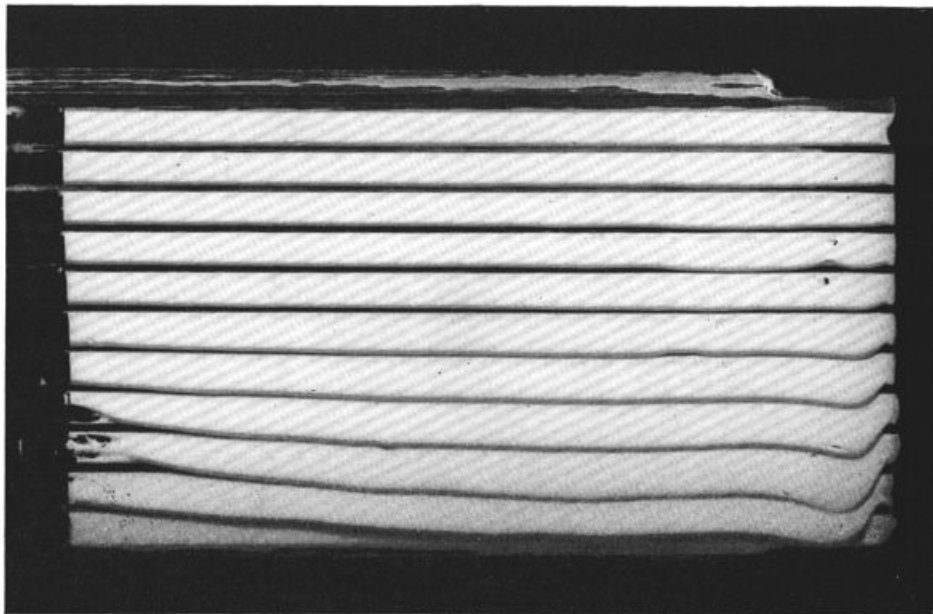


Figure 14. Typical sag test. This paste has a sag number of 5.

span of time taken to reach a given sag number in five successive trials on one paste.

CONCLUSIONS

The measurement and control of the complex and rather ill-defined rheological properties of semisolids are becoming increasingly important to industry and to the consumer to-day. It is hoped that the methods described will prove useful in factories and in other laboratories for research, development and quality control. In the absence of expensive, multipurpose equipment, adequate measurements may still be made with rather simple devices.

This work has been based on the research of scientists from the United Kingdom, South Africa, Israel and the United States on materials as diverse as cooking fats, butter, Napalm and paints.

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