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The evaluation of non-Newtonian viscosity using a modified parallel-plate plastometer

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A modified parallel-plate plastometer has been developed to measure the viscosity of rapid setting dental composite pastes. The theoretical treatment presented enabled the shear rates occurring in the specimen to be determined and, therefore, different materials to be compared. Five pastes were shown to be pseudoplastic power-law fluids each with unique K and n values.

1. Introduction

Rheology has considerable application in dentistry where the ease of mixing of components of dental materials and their subsequent handling is a rheological problem, and the success of a material depends, in part, on its ability to flow into all areas of a tooth cavity. Polymeric composite pastes, consisting essentially of dispersed inorganic particles (70% by volume) bound by a high molecular weight polymer [1], are used as tooth filling materials. The rheology of this group of materials is interesting since they are non-Newtonian, appear to have very low yield values after mixing and set rapidly. Previous workers have characterized their setting reactions empirically by recording consistency change with time [2, 3] and Braden [4] has studied the viscosity of component pastes using a cone and plate viscometer.

A modified parallel-plate plastometer has been designed to measure the viscosity of mixed, setting composite pastes [5]. This parameter is of clinical importance when considering the flow of a material during cavity filling. Conventional instruments are not suitable for the evaluation of these materials because specimen delivery is time consuming in a short setting reaction, and the fixed loading systems of existing parallel-plate instruments are not suitable for comparing materials whose viscosity is markedly different.

2. Experimental

2.1. The instrument

The plastometer consisted essentially of two horizontal stainless-steel platens (Fig. 1). The lower platen was mounted on a compression load-cell and the upper was attached to the cross-head of a universal testing machine.* The cross-head could be moved at a constant pre-set speed. The temperature of the platens was controlled by circulating water. A mechanism for specimen volume control at the centre of the lower platen enabled specimens 6 mm diameter and 3 mm high

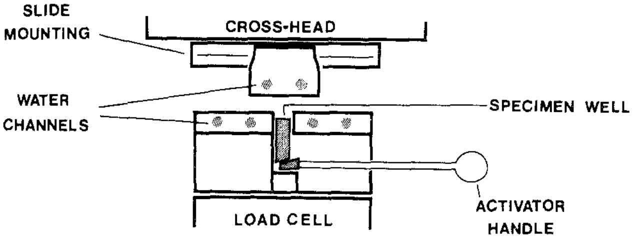

Figure 1 Schematic diagram of the parallel-plate plastometer. (Reproduced by kind permission of the Editor, *British Dental Journal*.)

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*Model 1195, Instron Ltd, High Wycombe, Bucks.

TABLE I Materials investigated

Material	Type	Batch nos. tested	Manufacturer
Adaptic	*bis*-phenol A-glycidal methacrylate composite	5K035	Johnson & Johnson, New Jersey
Cosmic	*bis*-phenol A-glycidal methacrylate/ urethane acrylate composite	SA11SB	Amalgamated Dental Manufacturing Co Ltd, London
Perfit	aromatic urethane acrylate composite	36/27	Allied Laboratories Ltd, London
Perfit H.V.	aromatic urethane acrylate composite	40/34	
Prestige	aromatic dimethacrylate composite	HPR0109	Lee Pharceuticals, California

to be positioned rapidly for testing by raising the floor of the well.

An extensometer was fitted across the two platens and calibrated so that the height of the specimen could be monitored accurately during each test. The output from the load cell was recorded simultaneously with this extensometer signal to give force/time and height/time curves.

2.2. Experimental procedure

Preliminary tests were carried out using coal tar pitch, a closely Newtonian material [6]. Four cross-head speeds were used, namely 2, 5, 10 and $20\,\mathrm{mm\,min^{-1}}$. Pitch has an extremely high viscosity at room temperature and so the tests were performed at 40 ± 0.5° C.

The main series of tests was on five dental composite pastes. These have the same basic formulation, but vary in the quantity and type of filler and monomer (Table I). The time available for manipulation after mixing is between 60 and 90 sec. Viscosity tests must be performed during the early stages of the manipulation time so that the increase in viscosity due to the curing reaction is negligible.

Before each test, the upper platen was set 2.5 mm above the lower. The material was mixed, inserted into the specimen well, and then the floor of the well was raised, thus positioning the specimen. The initial setting of the upper platen counteracted the 'doming' of the specimen that occurred when the specimen was extruded from the well 30 sec after the start of mixing, the upper platen was moved towards the lower. The movement was stopped when the specimen was 0.5 mm high.

A cross-head speed of $10\,\mathrm{mm\,min^{-1}}$ was used. For one material, (Adaptic) four cross-head speeds were used in order to assess the effect of a range of shear-rates. The tests were performed at 23 ± 0.5° C.

2.3. Theory

The theory of the parallel-plate plastometer is well developed [6–9]. The initial treatment presented here is based on Scott's work [8], and uses his solution for a generalized Newtonian fluid obeying a power-law viscosity function. A method of estimating the shear-rate distribution within the specimen is then presented.

The basic classical theory is derived from the Navier–Stokes equation which describes the motion of a Newtonian fluid:

$$\rho \dot{u} = \rho F - \nabla p + \eta \nabla^2 u$$

$$\nabla u = 0$$

where ρ is the density of the material, u is the velocity vector, F is the applied force, p is the fluid pressure, and η is the Newtonian coefficient of viscosity. For steady flow, and in the absence of body forces, this reduces to

$$\frac{\mathrm{d}p}{\mathrm{d}r} = \eta \left(\frac{\mathrm{d}^2 u_r}{\mathrm{d}z^2} + \frac{\mathrm{d}^2 u_r}{\mathrm{d}r^2} + \frac{1}{r}\frac{\mathrm{d}u_r}{\mathrm{d}r} \right)$$

$$\frac{\mathrm{d}p}{\mathrm{d}z} = \eta \left(\frac{\mathrm{d}^2 u_z}{\mathrm{d}z^2} + \frac{\mathrm{d}^2 u_z}{\mathrm{d}r^2} + \frac{1}{r}\frac{\mathrm{d}u_z}{\mathrm{d}r} \right)$$

where u_r is the radial velocity, and u_z is the velocity normal to the plates.

Scott [8], and Dienes and Klemm [7] then assumed that u_z was small compared with u_r and could, therefore, be neglected. This approximation is valid when the separation of the plates is small compared with their radius.

The equation therefore reduces to

$$\frac{\mathrm{d}p}{\mathrm{d}r} = \eta \frac{\mathrm{d}^2 u_r}{\mathrm{d}z^2}$$

$$z\frac{\mathrm{d}p}{\mathrm{d}r} = \eta \frac{\mathrm{d}u_r}{\mathrm{d}z}$$

Scott generalized this for a power-law fluid and allows for a yield stress to give

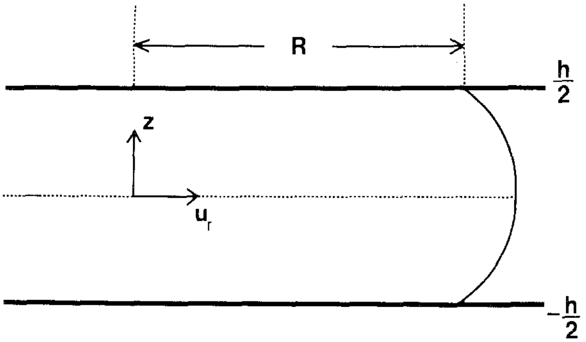

Figure 2 Diagram of the components of the radial velocity equation.

$$\left(z\frac{\mathrm{d}p}{\mathrm{d}r} - F\right) = K\left(\frac{\mathrm{d}u_r}{\mathrm{d}z}\right)^n.$$

He then uses this and a second hydrodynamic function, the momentum equation, to give an equation for the radial velocity (see Fig. 2).

$$u_r = \frac{(h/2)^{[1+(1/n)]}}{1+\frac{1}{n}}\left(-\frac{1}{K}\frac{\mathrm{d}p}{\mathrm{d}r}\right)^{1/n}\left[1-\left(\frac{2z}{h}\right)^{1+(1/n)}\right] \tag{1}$$

where h is the platen separation, K is the 'consistency', and n is the 'flow index'. This is then substituted into a third function, the mass balance equation, and integrated to give an equation for the applied force, F.

$$F = \frac{(\dot{h})^n}{h^{2n+1}}\left(\frac{2n+1}{n}\right)^n\frac{2^{(n+1)}\pi K R^{n+3}}{(n+3)}, \tag{2}$$

where R is the radius of the specimen.

For the type of plastometer in which the plates are large compared with the specimen, the condition V (the volume) $= \pi R^2 h$ may be inserted into Equation 2.

$$F = \frac{(\dot{h})^n}{h^{(5/2)(n+1)}}\left(\frac{2n+1}{n}\right)^n\frac{2^{(n+1)}\pi K}{n+3}\left(\frac{V}{\pi}\right)^{(n+3)/2} \tag{3}$$

In the present analysis, the rate of closure of the plates $(\dot{h})$ is constant. Equation 3 may, therefore, be plotted on logarithmic scales and a straight line would indicate either power-law or ideal Newtonian behaviour. The slope and intercept of the line would give values for n and K.

The viscosity of shear-rate dependent materials is usually expressed in terms of the apparent viscosity η_a which is the ratio of shear stress (τ) to rate of shear straining $(\dot{\gamma})$. For power law fluids, this is:

$$\eta_a = \frac{\tau}{\dot{\gamma}} = \frac{K\dot{\gamma}^n}{\dot{\gamma}} = K\dot{\gamma}^{n-1}.$$

The constants K and n are calculated from Equation 3. It therefore remains to determine the range of shear-rates imposed by each test.

The exact velocity profile within the specimen is dependent upon the material itself. This may be seen by inspection of Equation 1 where the z variation of u is governed by the flow index, n, of the material.

$$u_r(z) \propto \left[1-\left(\frac{2z}{h}\right)^{1+(1/n)}\right]$$

To simplify the problem, an approximation to the velocity profile may be made by assuming that the shear-rate varies only with time, i.e. that at a given time, there is a constant shear-rate across the specimen from $z=0$ to $z=h/2$. The velocity profile is then given by:

$$u_r(z) = u_c\left(1-\frac{2z}{h}\right),$$

where u_c is the velocity mid-way between the platens; then the shear-rate, $\dot{\gamma}$, is given by,

$$\dot{\gamma} = \frac{\mathrm{d}u_r}{\mathrm{d}z} = \frac{-2u_c}{h}. \tag{5}$$

The average velocity $u_c/2$ may be calculated as follows: The variables R and h are related by $V = \pi R^2 h$:

$$\frac{\mathrm{d}R}{\mathrm{d}h} = -\frac{1}{2}\sqrt{\left(\frac{V}{\pi}\right)}h^{-(3/2)}.$$

Therefore,

$$\frac{u_c}{2} = \frac{1}{2}\frac{\mathrm{d}R}{\mathrm{d}t} = \frac{1}{2}\frac{\mathrm{d}R}{\mathrm{d}h}\frac{\mathrm{d}h}{\mathrm{d}t} = -\frac{1}{2}\sqrt{\left(\frac{V}{\pi}\right)}h^{-(3/2)}\dot{h},$$

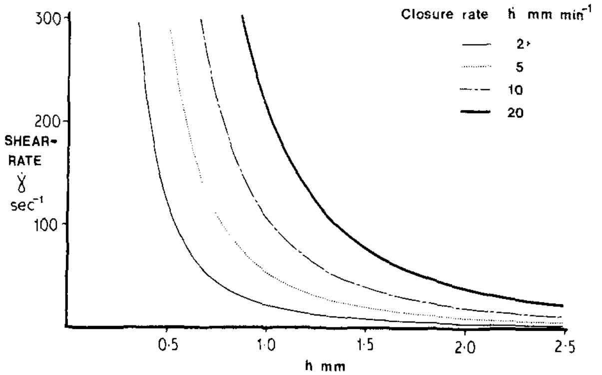

Figure 3 Variation of shear-rate with height of specimen at four rates of closure.

TABLE II Consistency (K) and flow index (n) values for the test materials

Material	$K \times 10^3$ ± S.D. ($\text{N m}^{-2}\,\text{sec}^{-1}$)	n ± S.D.
Adaptic	1.12 ± 0.13	0.63 ± 0.08
Cosmic	1.20 ± 0.13	0.25 ± 0.03
Perfit	0.115 ± 0.015	0.41 ± 0.05
Perfit H.V.	0.183 ± 0.024	0.50 ± 0.06
Prestige	3.21± 0.37	0.25 ± 0.03

then, from Equation 5,

$$\dot{\gamma} = 2\sqrt{\left(\frac{V}{\pi}\right)}\,h^{-(5/2)}\dot{h}. \tag{6}$$

This function is shown graphically in Fig. 3.

3. Results

The results for coal-tar pitch were analysed according to the Newtonian formula ($n = 1$, $K = \eta$, the Newtonian viscosity). The variation was essentially linear ($r > 0.99$) over a range of h within the limits $h = 2.2$ to 0.6 mm. There was no significant variation with platen displacement rate.

The logarithmic plots for the composites were accurately linear ($r < -0.99$) over a wide range (typically $h = 1.8$ to 0.6 mm) (Fig. 4). Values for K and n are shown in Table II.

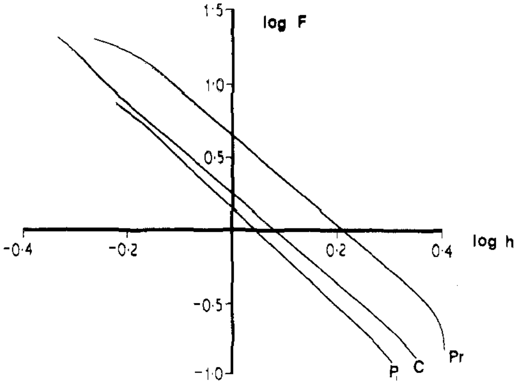

Figure 4 Typical log F/log h plots for three composites: P_1 – Perfit 'Normal', C – Cosmic, Pr – Prestige.

The variation of apparent viscosity with shear-rate for several platen displacement rates is shown in Fig. 5, and the variation for each material tested at one displacement rate is shown in Fig. 6.

4. Discussion

Analysis of the data for coal-tar pitch showed good agreement with the Scott Newtonian formula. This indicated that the modified plastometer was comparable with conventional plastometers for determining Newtonian viscosity.

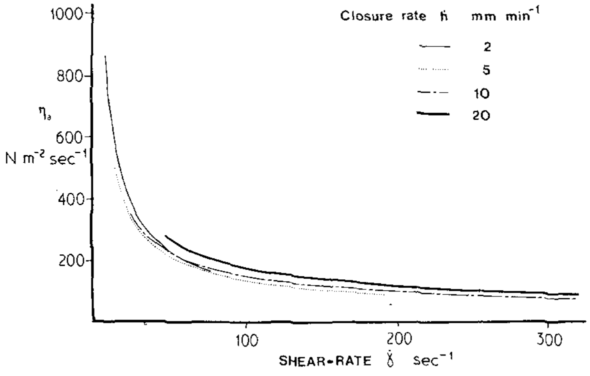

Figure 5 Variation of apparent viscosity with shear-rate for four rates of closure (Adaptic at 23 ± 0.5° C).

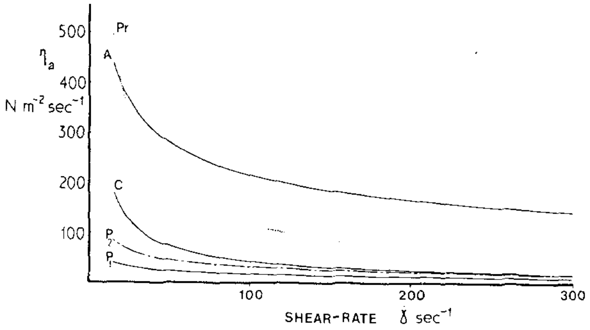

Figure 6 Relationship of apparent viscosity with shear-rate for five composites: Pr – Prestige; A – Adaptic; C – Cosmic, P_2 – Perfit 'H.V.'; P_1 – Perfit 'Normal'.

The composite pastes showed wide deviations from ideal Newtonian behaviour but were well described by Scott's general theory for materials with a power-law viscosity function. As the theory is valid for h small compared with R, agreement for $h > 2.00$ mm would not be expected.

The suggested method of estimating the shear-rates appears to give consistent results as the cross-head speed is varied (Fig. 5). As the approximation has less validity when n is small, there will be a greater error in the curves Pr and C in Fig. 6. However, it has so far been difficult to verify the analysis either by comparison with other instruments (none of which are suitable for this type of material) or by use of a 'standard' material (other high viscosity fluids are commonly Bingham fluids, or show appreciable elastic effects). A knowledge of the shear-rates is important if a full rheological comparison between materials is to be made.

Braden [4] suggests that shear failure occurs at very low shear-rates in this group of materials, which was manifested in the cone and plate viscometer by material breaking away from the plates and by departures from linearity on the shear stress–shear-rate plots. The present authors considered the possibility of shear failure occurring at the shear-rates reported here, but found consistent and reproducible flow behaviour throughout the series of experiments. Furthermore, Hutton [10], in a study of fracture of silicone liquids, states that the occurrence of shear failure will depend on the geometry of the device in which the liquid is sheared. The occurrence of shear failure in the polymeric composites merits further study.

The modified plastometer has an important advantage over conventional instruments in that materials of different viscosity can be compared without altering the experimental dynamics. This is because the shear-rates induced are largely independent of the material under test.

There are also a number of practical advantages important to the dental application. High shear-rates may be imposed which are probably of the same order as those induced by the dentist during cavity filling and the wide difference in the viscosity of dental composites does not complicate the technique. The rapid operation and cleaning of the instrument render it suitable for analysing setting materials.

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