A study of the rheological properties of endodontic sealers

S. Lacey, T. R. Pitt Ford, T. F. Watson & M. Sherriff

Department of Biomaterials and Conservative Dentistry, GKT Dental Institute, King's College London, London, UK

Abstract

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Aim To test the hypothesis that there would be no statistically significant difference in viscosity-related measures of endodontic sealers or change in these with strain rate, internal diameter or powder : liquid ratio in a capillary system.

Methodology Materials used were Apexit, Tubliseal EWT, Grossman's sealer and Ketac-endo. Viscosity-related measures were tested in a two-plate test, and in a capillary rheometer. The mean values ($n = 12$) for thickness and diameter of material formed between two glass plates were tested with one-way analysis of variance. Pressure was applied to a capillary rheometer at strain rates 5 and 10 mm min^{-1} in tubes of internal diameter 0.6 and 1.2 mm.

Results Tubliseal EWT had a thinner film thickness than the other sealers ($\alpha = 0.05$). The difference in diameter between Tubliseal EWT and the other sealers was significant apart from Apexit. Increased strain rate gave a significant increase ($\alpha = 0.05$) in the flow of all sealers. Narrower tubes produced increased velocity, which was significant for all sealers, and reduced volumetric flow, which was significant for all sealers except Grossman's 2 : 1 (Wilcoxon signed rank test). Reduction in powder : liquid ratio of Grossman's significantly increased flow in narrow tubes and at higher strain rate (Mann–Whitney test).

Conclusion There was a significant difference between the flow of Tubliseal EWT and the other sealers tested in the two-plate test; capillary flow was affected by sealer, internal diameter, strain rate and powder : liquid ratio. The null hypotheses were rejected.

Keywords: capillary rheometer, endodontic sealers, rheology.

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Introduction

Successful root canal treatment depends on the thorough debridement of the root canal system, the elimination of pathogenic organisms and finally the complete obturation of the canal (Sundqvist & Figdor 1998). Successful obturation is achieved by the adhesion and stability of the sealer to the canal walls which, amongst other factors, is affected by its rheological properties. The ideal properties of a root canal sealer are those which provide ease of handling for the clinician, enable obturation without voids to the apex, produce a permanent biocompatible seal and can be readily removed if necessary for retreatment. Furthermore, it should be impervious to tissue fluids, should not discolour tooth tissue, and be radiopaque (Branstetter & von Fraunhofer 1982, Buck 2002). However, these ideal properties will be less relevant if the sealer does not flow to the apex and into all internal irregularities (Wolcott *et al.* 1997). In this case there will be residual space for existing pathogenic bacteria to remain viable and the possibility of two-way leakage of bacteria, nutrients and bacterial toxins.

The ISO and ADA specifications for flow of endodontic sealers have not required a measurement of viscosity; they use the diameter of a film of sealer between

Correspondence: Susanna Lacey, Department of Biomaterials and Conservative Dentistry, GKT Dental Institute, King's College London, Guy's Hospital, London Bridge, London SE1 9RT, UK (Tel.: +44(0)207 188 1594, fax: +44(0)207 188 1583; e-mail: susanna.lacey@kcl.ac.uk).

two glass plates (ANSI/ADA 2000, BS EN ISO 6876 2002) which is related to viscosity but easier to measure.

As yet, no root canal sealer has all the ideal properties and most leak with time, either through poor initial adaptation to the canal walls or due to solubility and disintegration of the sealer (Branstetter & von Fraunhofer 1982, Hovland & Dumsha 1985, Kersten & Moorer 1989, Behrend *et al.* 1996, Spangberg 1998). Ideal flow requirements have not yet been established that will allow the material to flow into all internal spaces, without extrusion through the apex, producing a seal, which is closely adapted to the canal walls, and without voids. A study of the rheological properties of endodontic sealers may help to achieve an ideal flow pattern.

The aim of this study was to compare the viscosity of four commercially available endodontic sealers using viscosity-related measures of film thickness and diameter in a two-plate system and velocity and volumetric flow rate in a custom-made capillary rheometer system. The null hypothesis was that there would be no significant difference ($\alpha = 0.05$) in the viscosity-related measures of endodontic sealers. Secondly, the aim was to investigate the effect of increased shear rate and reduced internal diameter on flow, using volumetric flow and velocity as the viscosity-related measures in a capillary system. The null hypothesis for this element of the study was that there would be no significant difference ($\alpha = 0.05$) in the viscosity-related measures of endodontic sealers with increased shear rate or reduced internal diameter. Thirdly, the aim was to quantify the effect on velocity and volumetric flow of varying powder : liquid ratio of Grossman's sealer, the null hypothesis being that there would be no significant ($\alpha = 0.05$) difference in viscosity-related measures with change in powder : liquid ratio from 3 : 1 to 2 : 1.

Materials and methods

The materials are listed in Table 1. They were freshly mixed for each experiment. The materials were chosen to be representative of zinc oxide eugenol, calcium hydroxide and glass–ionomer sealers. Two ratios of Grossman's (3 : 1 and 2 : 1 powder : liquid by weight) were chosen to investigate the viscosity effects produced by varying the powder : liquid ratio.

Rheological properties of the sealers were studied using two methods:

1. two-plate method;

2. custom-made capillary rheometer.

Two-plate method

The experiments were carried out at room temperature (23 ± 2 °C). Square glass plates were produced to required specifications by a glazier. The dimensions were 3 mm thick, 4.5 mm square for the base and 3.8 mm square for the cover. The thickness of each plate was measured by micrometer. A volume of 0.1 mL of each material was delivered via a 1 mL syringe onto the base plate, covered with the top plate, a 30 g weight was applied for 30 s then removed. The resultant thickness was measured by micrometer and the film thickness of the sealer obtained by subtracting the combined original thickness of the plates. The diameter of the film of sealer was measured microscopically (Graticules, Tonbridge, UK) and the mean taken of two readings at right angles to each other. A small pilot study of three samples of each material was initially carried out to determine the minimum sample size required to establish if there was a significant difference in the sealers. The significance was set at $\alpha = 0.05$. The sample size, calculated from the formula for hypothesis testing at 90% power (Kirkwood 1997), was 12 samples for each material.

Capillary rheometer

Flow in fine capillary tubes was selected as a suitable model for assessing flow in root canals. The custom-made capillary rheometer (Fig. 1) was constructed in the laboratory using glass capillary tubes of known length (150 mm) and internal diameter (Harvard Apparatus, Edenbridge, UK), plastic tubing (Tygon, Corby, UK) and luer attachments (Sims Portex Ltd,

Table 1 Materials used, their manufacturers, presentation and chemical type

Material	Manufacturer	Presentation	Chemical type
Apexit	Ivoclar Vivadent, Schaan, Liechtenstein	Paste/paste	Calcium hydroxide
Tubliseal EWT	Kerr, Romulus, MI, USA	Paste/paste	Zinc oxide eugenol
Grossman's sealer	Guy's and St Thomas' NHS Trust, London, UK	Powder/liquid	Zinc oxide eugenol
Ketac-endo	3M-ESPE, Seefeld, Germany	Capsule	Glass–ionomer

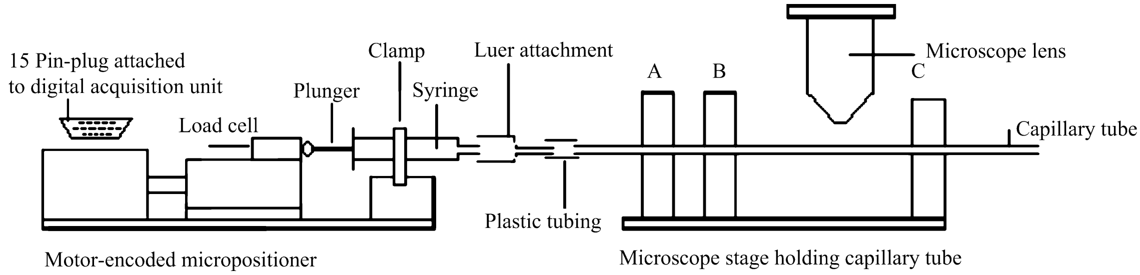

Figure 1 A custom-made capillary rheometer for applying pressure at a constant rate to sealer in a capillary tube.

Hythe, UK), 1 mL syringes (Terumo Syringes, Leuven, Belgium), a custom-made brass plate manufactured to support the capillary tube and to fit onto the microscope stage and a motor-encoded micropositioner (Physik Instrumente, Karlsruhe, Germany), which applied a load at constant rate to the plunger of the syringe. The time taken for the material to flow between fixed points (A, B and C) on the capillary tube was measured using a stopwatch. The distance A–B was 32 mm and B–C was 37 mm. Equal values obtained for velocity between these points would indicate that a steady-state velocity profile had been achieved. The loads were applied by a DC motor-encoded micropositioner and their values recorded on a digital acquisition unit. The measurements were taken at constant rates of 5 and 10 mm min^{-1}, using tubes of internal diameter 0.6 and 1.2 mm. As this was the first use of the apparatus, a sample size could not be calculated from previous pilot studies. It was decided to take five samples for each material at each strain rate and for each internal diameter of the capillary tubes. The room temperature and relative humidity were recorded at each reading to ensure that the temperature was within the range of 23 ± 2 °C. The relative humidity during the experiments, taken from a laboratory barometer, varied from 50 to 64%. It was considered that within the relatively closed capillary system of the apparatus the effect of variations in humidity would be negligible. The volumetric flow rate and velocity of each material were calculated for each flow category of strain rate and internal diameter.

Statistical analysis

Twelve samples for each material were taken for the viscosity-related measures of film thickness and diameter. The arithmetic means and 95% confidence intervals were calculated for each. For statistical analysis of difference between each material, one-way analysis of variance (ANOVA) was used and the significance level was set at $\alpha = 0.05$. *Post-hoc* tests were run for unequal variances (SPSS 11.5; SPSS Inc., Chicago, IL, USA).

In the capillary rheometer experiments five samples were taken for each material at each strain rate and in tubes of both internal diameters. The mean and 95% confidence intervals of velocity and volumetric flow were calculated for each. The values ($n = 5$) of velocity and volumetric flow across the two lengths of capillary tube (A–B and B–C) were reduced to an integer and tested for equality using DELTA function (Microsoft Excel). The means for velocity and volumetric flow for each sealer were tested for significant difference ($\alpha = 0.05$) with Kruskal–Wallis (SPSS 11.5) at each flow category. The percentage change in velocity and volumetric flow with increased strain rate and reduced internal diameter was calculated for each sealer at each flow category and tested for significant difference using the Wilcoxon signed rank test (SPSS 11.5). The percentage change in volumetric flow and velocity of Grossman's sealer on varying powder : liquid ratio from 3 : 1 to 2 : 1 was also calculated and tested for significant difference using the Mann–Whitney test (SPSS 11.5).

Results

Two plates

The means, standard deviations (SD) and 95% confidence intervals (CI) for diameter and film thickness of the endodontic sealers are shown in Table 2, with Tubliseal EWT displaying the least film thickness and greatest diameter. There was a significant difference in film thickness between Tubliseal EWT and the other sealers (ANOVA: $\alpha = 0.05$) but no significant differences between the other sealers. There was a significant difference between the diameter of Tubliseal EWT and Ketac-endo, Grossman's 3 : 1 and 2 : 1 but not Apexit. There was no significant difference between the diameters of the other sealers.

Table 2 Mean film thickness and diameters of endodontic sealers ($n = 12$) with standard deviation (SD) and 95% confidence interval (CI)

Sealer	Film thickness (mm)	SD	95% CI	Diameter (mm)	SD	95% CI
Apexit	0.280	0.06	0.323–0.236	24.744	3.4	26.68–22.80
Grossman's 2 : 1	0.260	0.05	0.274–0.239	22.294	1.7	22.63–21.96
Grossman's 3 : 1	0.254	0.03	0.283–0.224	23.282	0.6	24.22–22.34
Ketac-endo	0.287	0.05	0.322–0.252	22.350	0.9	22.94–21.76
Tubliseal EWT	0.126	0.04	0.150–0.101	25.973	1.4	26.77–25.17

Table 3 Mean velocity (V) and volumetric flow (Q) of endodontic sealers at rates 5 and 10 mm min^{-1} and internal diameter (ID) 1.2 and 0.6 mm showing standard deviations (SD) and 95% confidence intervals (CI). Measurements taken between points A and B and B and C in capillaries 150 mm long ($n = 5$)

			Volumetric flow rate (Q, mm^3 s^{-1})						Velocity (V, mm s^{-1})					
	ID	Rate	A–B	SD	95% CI	B–C	SD	95% CI	A–B	SD	95% CI	B–C	SD	95% CI
Apexit	1.2	5	1.94	0.29	2.2–1.69	1.9	0.2	2.08–1.72	1.61	0.27	1.85–1.38	1.68	0.18	1.92–1.45
	1.2	10	2.86	0.33	3.15–2.57	2.83	0.38	3.16–2.5	2.52	0.29	2.78–2.27	2.5	0.33	2.79–2.22
	0.6	5	0.99	0.09	1.07–0.91	0.98	0.35	1.29–0.68	3.5	0.33	3.79–3.21	3.48	1.23	4.55–2.4
Tubliseal EWT	1.2	5	1.76	0.83	2.49–1.04	1.62	0.32	1.89–1.33	1.54	0.75	1.78–1.31	1.42	0.28	1.67–1.92
	1.2	10	2.77	0.2	2.95–2.6	2.88	0.12	2.98–2.77	2.45	0.18	2.61–2.29	2.54	0.11	2.64–2.45
	0.6	5	0.92	0.1	1–0.83	0.88	0.1	0.97–0.79	3.24	0.36	3.55–2.92	3.11	0.35	3.42–2.81
Ketac-endo	1.2	5	1.32	0.16	1.46–1.18	1.41	0.21	1.59–1.22	1.15	0.14	1.3–0.92	1.24	0.18	1.48–1.01
	1.2	10	3.55	1.08	4.26–2.4	2.91	0.11	3–2.81	2.94	0.94	3.77–2.11	2.57	0.1	2.6–2.49
	0.6	5	1.01	0.45	1.4–0.61	1.09	0.56	1.58–0.6	3.57	1.6	4.97–2.17	3.87	1.99	5.61–2.12
Grossman's 3 : 1	1.2	5	1.42	0.28	1.66–1.17	1.43	0.26	1.66–1.21	1.22	0.27	1.46–0.98	1.27	0.23	1.51–1.03
	1.2	10	4.32	1.82	5.91–2.72	3.6	0.55	4.08–3.11	3.82	1.61	4.35–3.28	3.18	0.49	3.61–2.75
	0.6	5	0.85	0.18	1.01–0.69	0.78	0.2	0.96–0.61	3.01	0.65	4.3–1.72	2.8	0.71	4.27–1.28
Grossman's 2 : 1	1.2	5	1.44	0.14	1.56–1.32	1.45	0.13	1.57–1.34	1.24	0.15	1.48–1.01	1.29	0.12	1.52–1.05
	1.2	10	3.02	0.45	3.41–2.62	3.12	0.31	3.39–2.84	2.67	0.4	3.02–2.32	2.76	0.28	3–2.51
	0.6	5	1.32	0.32	1.6–1.04	1.61	0.57	2.11–1.11	4.66	1.14	5.66–3.66	5.68	2.01	7.44–3.92

Capillary rheometer

Table 3 shows the mean volumetric flow rate (Q) and the velocity (V) of all sealers at both strain rates (γ) and in tubes of both internal diameters (ID). SD and 95% CI for volumetric flow and velocity are also shown. The values for volumetric flow rate and velocity for flow from A to B and B to C showed an overall equality of 84% using DELTA function (Microsoft Excel). It can be interpreted that the apparatus was producing steady-state flow. The results of the Kruskal–Wallis test are shown in Table 4. There was a significant difference in the flow of all sealers at all flow categories. The percentage changes in volumetric flow and velocity with increased strain rate and reduced internal diameter are shown in Table 5. The results of the Wilcoxon signed rank test for significance of these changes are also shown. On increasing the strain rate from 5 to 10 mm min^{-1}, there was a significant increase ($\alpha = 0.05$) in the flow of all sealers. On reducing the internal diameter from 1.2 to 0.6 mm, there was increased velocity, which was significant for all sealers, and decreased volumetric flow, which was significant for all sealers except Ketac-endo and Grossman's 2 : 1.

Table 4 Kruskal–Wallis test for significant difference in velocity and volumetric flow between sealers at each flow category

	Velocity			Volumetric flow		
	1.2/5	1.2/10	0.6/5	1.2/5	1.2/10	0.6/5
χ^2	14.561	18.292	17.253	17.273	18.774	14.319
d.f.	4	4	4	4	4	4
P	0.006	0.001	0.002	0.002	0.001	0.002

Grouping variable: sealer.

Table 5 Percentage change in volumetric flow and velocity with probability results of Wilcoxon signed rank test

	Increased strain rate	*P*	Reduced internal diameter	*P*
Percentage change in volumetric flow				
Apexit	48	0.005	−48	0.005
Tubliseal EWT	67	0.007	−47	0.005
Ketac-endo	129	0.012	−23	0.241
Grossman's 3 : 1	177	0.005	−43	0.005
Grossman's 2 : 1	112	0.005	−1	0.139
All sealers	101	0.000	−34	0.000
Percentage change in velocity				
Apexit	53	0.005	−112	0.005
Tubliseal EWT	68	0.007	−113	0.007
Ketac-endo	130	0.005	−210	0.005
Grossman's 3 : 1	182	0.005	−134	0.005
Grossman's 2 : 1	115	0.005	−310	0.005
All sealers	104	0.000	−169	0.000

Table 6 Percentage change in flow on changing powder : liquid ratio of Grossman's from 3 : 1 to 2 : 1 ($n = 10$)

Flow category (internal diameter/strain rate)	Volumetric flow	P^a	Velocity	P^a
1.2/5	2.6	0.074	2.98	0.184
1.2/10	−18.34	0.019	−18.3	0.019
0.6/5	85.5	0.002	85.8	0.001

[a]Mann–Whitney test.

The percentage difference in volumetric flow and velocity of Grossman's 3 : 1 and 2 : 1 with probability values at each flow category are shown in Table 6. There was slight increase in flow (2.6 and 2.9%) with reduction in powder : liquid ratio at strain rate 5 mm min^{-1}. This effect was much greater in the narrower tubes (85.5 and 85.8%). At higher strain rate there was reduced flow (18.3%) with reduction in powder : liquid ratio. These changes were significant ($\alpha = 0.05$) in the narrow tubes and at higher shear rate.

Discussion

The two-plate method is a simple procedure to carry out, giving viscosity-related measures. Using this method, variation in the flow of endodontic sealers has been shown previously (McComb & Smith 1976, Ørstavik 1982, Ono & Matsumoto 1998, Mendonca *et al.* 2000, Siqueira *et al.* 2000). This study has demonstrated that Tubliseal EWT has significantly lower viscosity and therefore significantly better flow than the other sealers at 23 ± 2 °C. With the capillary system, however, the sealer showing the most flow as demonstrated by volumetric flow and velocity, varied for each flow category. At the base flow category of this model system (1.2 mm internal diameter and strain rate 5 mm min^{-1}), Apexit had the most flow, and Ketac-endo the least. At a higher strain rate of 10 mm min^{-1} Grossman's 3 : 1 had the most flow, and Tubliseal EWT the least, whereas in the narrower tubes Grossman's 2 : 1 had the most flow and Grossman's 3 : 1 the least flow. These measurements should be taken while the fluid is in laminar rather than turbulent flow. In addition to checking the velocity between points A–B and B–C, ensuring a minimum ratio of tube length to tube diameter of 100 : 1 avoids entry and exit effects, and gives laminar flow (Zahler & Murfitt 1963, Walters 1975, Barnes 2000). The tubes used in these experiments were all 150 mm long.

The measurement of flow of setting cements may be made complex by the setting process, where the development of chemical cross-linking chains affects the viscosity (Barnes *et al.* 2001). This problem was avoided by taking all measurements within the manufacturer's recommended working time. A mixing technique, according to the manufacturer's instructions, was adhered to, and the time from the start of mix to the end of test was noted for each material (<3 min).

The custom-made rheometer, which was constructed from materials easily obtained in the laboratory, provided a better testing method for sealers than that recommended by the BS EN ISO 6876 (2002) specification as it can demonstrate the probable effect of narrowing of root canals or increasing the rate of insertion. There was the advantage of using a relatively small amount (0.3 mL) of sealer material. Previous studies have shown that flow depends on particle size (Weisman 1970), rate of shear (Uhrich *et al.* 1978), temperature and time (Vermilyea *et al.* 1978, Watts *et al.* 1981). This study has, in addition, demonstrated the rheological fact that in a capillary system, flow depends on the internal diameter of the tubes and the rate of insertion, and that this effect varies with different materials.

The rheology of endodontic sealers may be complicated by tapered and curved canals and by the insertion of a gutta-percha point. Flow may be affected by dentine tubules and a smear layer. However, the results from the capillary system would appear to relate more closely to the clinical situation than the two-plate method. Viscosity, and therefore resistance to flow, is inversely related to eight times the volumetric flow rate (Poisseuille equation). This study has shown that increasing the rate of insertion gives increased volumetric flow and therefore reduced viscosity for all sealers. As the internal width of the canals is reduced, there is reduced volumetric flow and therefore increased viscosity. Clinicians may have assumed that flow of sealers will be improved by increasing the rate of insertion. However, it is necessary to consider not only which sealer is used but also the width of the root canals. Nor can it be assumed that reducing the powder : liquid ratio of a sealer will lead to improved flow, as this study has shown that flow may be reduced at a higher rate of insertion.

Conclusion

There was a significant difference between the flow of Tubliseal EWT and the other sealers tested in the two-plate test; capillary flow was affected by sealer, internal diameter, strain rate and powder : liquid ratio. The null hypotheses were rejected.

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