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The effect of temperature on viscosity of root canal sealers

S. Lacey[1], T. R. Pitt Ford[1], X.-F. Yuan[2], M. Sherriff[1] & T. Watson[1]

[1]Department of Biomaterials and Conservative Dentistry, Dental Institute, King's College London, London; and [2]School of Chemical Engineering and Analytical Science, University of Manchester, Manchester, UK

Abstract

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Aim To test the hypothesis that there was no significant ($\alpha = 0.05$) change in viscosity of commercially available root canal sealers with increase in temperature using a high-performance Advanced Rheometric Expansion System (ARES) rheometer.

Methodology Materials tested were Apexit, Tubliseal EWT, Grossman's, AH Plus and Ketac-endo. Cone-and-plate geometry was used (25-mm diameter, 0.1 radian and gap 0.051 mm). Measurements were carried out for steady-state viscosity at 25 and 37 °C in the shear rate range of 0.001–50 s^{-1} at standardized relative humidity and within 30 min from the start of mixing. Five samples were taken for each sealer at each temperature.

Results At 25 °C all sealers demonstrated shear thinning. At 37 °C Grossman's (powder : liquid ratio 2 : 1 and 3 : 1) and Ketac-endo had a rapid rise in viscosity and early set whereas the other sealers were shear thinning. On increasing temperature from 25 °C to 37 °C, Apexit, Tubliseal and AH Plus had reduced viscosity whereas Grossman's 2 : 1, Grossman's 3 : 1 and Ketac-endo had increased viscosity, which varied with the shear rate. The change in viscosity with change in temperature was significant ($P < 0.05$) for all sealers except AH Plus.

Conclusions There was a variation in the effect of increasing temperature on each sealer depending on the shear rate. With the exception of AH Plus, a significant ($P < 0.05$) change in viscosity was found, and the null hypothesis was rejected.

Keywords: cone-and-plate rheometer, root canal sealer, viscosity.

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Introduction

The filling of root canals relies on the use of root canal sealers, which need to flow during the process into canal irregularities and adapt to the walls. Flow properties are influenced by the viscosity of the material, temperature and humidity. They are also likely to be affected by the shape and width of the canal. ISO specifications for flow of sealers require that when placed between two glass plates at 23 ± 2°C and a known weight applied for a fixed time, the resultant film should have a diameter of at least 20 mm (BS EN ISO 6876 2002). Various types of rheometer have been used to study the flow properties of sealers and other dental materials in which the effects of temperature (Braden 1967, Plant *et al.* 1972), measurement time (Uhrich *et al.* 1978, Ørstavik 1983), shear rate (Vermilyea *et al.* 1979, Ferracane *et al.* 1981) and additives (Hill & Wilson 1988) have been investigated. After mixing at room temperature and introducing into the root canal, root canal sealers are subjected to temperature change from ambient to mouth temperature. In some filling techniques, further heat may then be applied. For simple Newtonian fluids, viscosity decreases with increase in temperature. For complex fluids such as setting pastes the effect of temperature may be much more pronounced (Barnes 2002). In previous work on sealers, a custom-made capillary rheometer was used to study the possible effect of change in shear rate and internal diameter of the root

Correspondence: Dr Susanna Lacey, Dental Institute, Floor 25, Guy's Hospital, London SE1 9RT, UK (Tel.: 0207 188 1162; fax: 0207 188 1159; e-mail: susanna.lacey@kcl.ac.uk).

canal (Lacey *et al.* 2005). However, little work has been done on the effect of temperature on the rheological properties of sealers.

The aim of this study was to characterize some commercially available root canal sealers across clinically relevant shear rates and to investigate the effect of temperature on the viscosity of different sealers. The null hypothesis was that there would be no significant difference ($\alpha = 0.05$) in the viscosity of endodontic sealers with increase in temperature from 25 °C to 37 °C.

Materials and methods

Rheological characterization was carried out using a high-performance strain-controlled advanced rheometric expansion system (ARES) rheometer (Rheometric Scientific Ltd, Piscataway, NJ, USA) with cone-and-plate geometry (cone angle = 0.1 radian and cone diameter = 25 mm), and Peltier plate-temperature control (±0.1 °C). The cone-and-plate gap between the central points of the stationary cone and the rotating plate was set at 0.051 mm (Fig. 1). Materials used were Apexit (Ivoclar Vivadent, Schaan, Lichtenstein), Tubliseal EWT (Kerr, Romulus, MI, USA), Grossman's sealer (Guy's and St Thomas NHS Trust, London, UK) at powder : liquid ratios, 3 : 1 and 2 : 1 by weight, AH Plus (Dentsply, Weybridge, UK) and Ketac-endo (3M Espe, Seefeld, Germany). These materials with manufacturers' names, batch numbers, chemical type and delivery system are listed in Table 1.

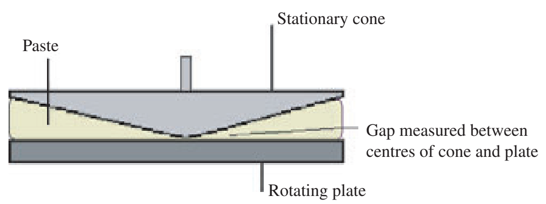

Figure 1 Cone-and-plate geometry.

For each experiment, the materials were freshly mixed according to the manufacturers' instructions. The experiments were carried out at 25 °C (room temperature) and 37 °C (mouth temperature) for each material with the shear rate range from 0.001 to 50 s^{-1}. Each experiment was completed within 30 min from start of mixing. The effect of variations in relative humidity was minimized by ensuring that there was excess material beyond the outer rim of the cone. Five samples were taken for each material at each temperature. Graphs were produced of the viscosity of each material at both temperatures across the range of shear rates. The viscosity versus shear rate curves at constant temperature were obtained from the steady rate sweep test and allowed 30 s measurement time at each shear rate. Overlays of these graphs were drawn for each temperature (Fig. 2). On one day, one series of experiments was run for each material at both the temperatures and again the graphs were produced. The mean viscosity and standard deviation at three shear rates (0.05, 0.1 and 1 s^{-1}) were calculated for each material at both the temperatures (Table 2). The percentage change in viscosity was calculated for each sealer. The change in viscosity for each sealer was tested for significant difference ($\alpha = 0.05$) using Wilcoxon signed rank test (SPSS 13.5; SPSS Inc., Chicago, IL, USA). For those materials which had set at 37 °C, the value for viscosity was taken as that at the top of the scale on the chart.

Results

Figure 2 shows an overlay of the graphs obtained for the viscosity of all sealers across the shear rate range from 0.001 to 50 s^{-1}, at 25 °C and 37 °C. At 25 °C the initial viscosity ranged from 5000 Pa-s for AH Plus to 100 000 Pa-s for Grossman's 3 : 1. All sealers demonstrated shear thinning, i.e. decrease in viscosity with increase in shear rate.

At 37 °C, the initial viscosity ranged from 6000 Pa-s for Apexit to 100 000 Pa-s for Grossman's 3 : 1. For

Table 1 Materials used

Material	Manufacturer	Presentation	Chemical type	Batch no.
Apexit	IvoclarVivadent, Schaan, Lichtenstein	Paste/paste	Calcium hydroxide	D66305 A/D64006 B
Tubliseal EWT	Kerr's Romulus, MI, USA	Paste/paste	Zinc oxide/eugenol	25903
Grossman's sealer (P : L ratio 3 : 1, 2 : 1)	Guy's and St Thomas NHS Trust, London, UK	Powder/liquid	Zinc oxide/eugenol	DZY 124/070
Ketac-endo	3 M Espe, Seefeld, Germany	Capsule (P/L)	Glass–ionomer	2005–04
AH Plus	Dentsply, Weybridge, UK	Paste/paste	Epoxy-amine resin	606.20.110

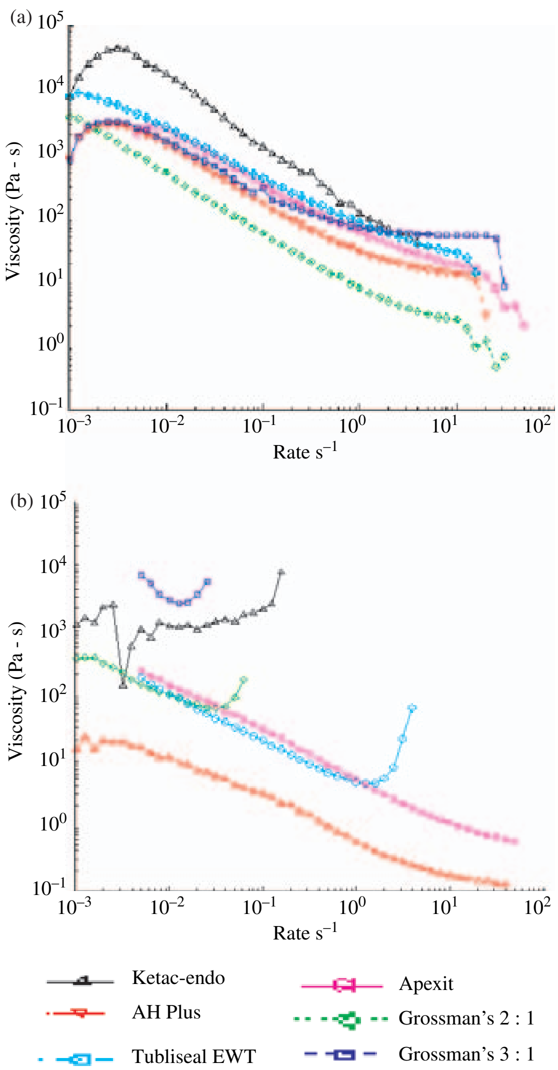

Figure 2 Overlays of graphs of viscosity of root canal sealers plotted against shear rate at 25 °C (a) and 37 °C (b).

Ketac-endo and both powder : liquid ratios of Grossman's there was an initial reduction in viscosity followed by a rapid increase in viscosity and early set, whereas Apexit, AH Plus and Tubliseal had continued shear thinning, with Tubliseal setting before the end of the experiment. Table 2 shows the mean viscosity ($n = 5$) of all sealers at three shear rates at both the temperatures. The sealers demonstrated differing flow patterns with increase in temperature. Apexit, Tubliseal EWT and AH Plus showed reduced viscosity with increased temperature at all shear rates (Fig. 3). Ketac-endo showed increased viscosity with increase in temperature. At the higher temperature, between strain rates 0.05 and 0.1 s^{-1} Ketac-endo showed Newtonian behaviour with no change in viscosity (Fig. 4); at shear rate 1 s^{-1} the material had set. Grossman's 3 : 1 showed increased viscosity with increased temperature but increasing the shear rate from 0.05 to 0.1 s^{-1} still had a shear-thinning effect (Fig. 5). However, there was also an earlier set so that at strain rate 1 s^{-1} there was no further flow. With increased temperature Grossman's 2 : 1 also showed increased viscosity which increased with shear rate (i.e. shear-thickening), so that at a rate of 1 s^{-1} there was no further flow (Fig. 6).

At each shear rate, statistical analysis of the results using Wilcoxon signed rank test (SPSS 13.5) showed significant difference ($P < 0.05$) in viscosity with increase in temperature for Apexit, Grossman's and Ketac-endo. The change in viscosity in Tubliseal EWT and AH Plus was not significant. When viscosity values at each shear rate were combined, only AH Plus did not show a significant difference ($\alpha = 0.05$) with increased temperature. The percentage change in viscosity with increased temperature and the rheological behaviours of shear thinning, shear thickening (dilatant) or Newtonian are also shown in Table 2.

Discussion

Cone-and-plate geometry was chosen in this study of endodontic sealers because it gives nearly homogeneous shear rate throughout the test material. The results thus obtained are most reliable. These are best for the characterization of the intrinsic properties of materials irrespective of their clinical use (McKennel 1960). Samples of each material used were all from the same batch. A standard mixing technique was used and materials weighed with a laboratory balance to the nearest milligram. However, there was still variation in the results as shown by the wide standard deviations. These might be explained by zero errors (when zero readings do not correspond to zero stress or strain rate) but this is unlikely with the level of accuracy of this apparatus.

Manufacturer's recommendations vary for each sealer. 'Long working times' for Apexit are given at 'normal temperature and humidity', while for Tubliseal EWT the time quoted (20–60 min) is at 21–27 °C. Working time for Ketac-Endo is given as 23 min at 23 °C and 50% humidity, while at 36 °C and 100% humidity, it is given as 7 min. AH Plus has a working time of 4 h at 23 °C. The working time of Grossman's may be prolonged by mixing on a chilled slab.

Table 2. Viscosity and rheological behaviour of root canal sealers at 25 °C and 37 °C and shear rates, 0.05, 0.1 and 1 s^{-1} with standard deviation values and percentage change with increase in temperature ($n = 5$)

Sealer		Temperature 25 °C		Rheological behaviour	Temperature 37 °C		Rheological behaviour	% change	Wilcoxon Signed Rank probablity	all rates combined $n = 15$
	rate	viscosity	SD		viscosity	SD				
Apexit	0.05	515.49	164.40	shear thinning	261.13	210.83	shear thinning	49% reduced	p = 0.04	P = 0.001
	0.1	320.53	93.18		100.86	99.76		69% reduced	p = 0.04	
	1	61.95	19.97		26.14	20.35		58% reduced	p = 0.04	
Tubliseal EWT	0.05	531.20	200.73	shear thinning	253.72	211.78	shear thinning	52% reduced	p = 0.08	P = 0.005
	0.1	339.66	124.62		146.09	121.99		57% reduced	p = 0.08	
	1	69.62	28.53		27.34	22.39		61% reduced	p = 0.08	
Grossman's 2:1	0.05	142.94	217.19	shear thinning	446.76	339.20	shear thickening	213% increased	p = 004	P = 0.001
	0.1	38.97	35.57		2170.92	2924.50		5471% increased	p = 0.04	
	1	5.16	5.88		set			set	p = 0.04	
Grossman's 3:1	0.05	497.99	736.75	shear thinning	set		shear thickening	set	p = 0.04	P = 0.001
	0.1	324.63	496.10		set			set	p = 0.04	
	1	105.90	32.59		set			set	p = 0.11	
Ketac-endo	0.05	1042. 53	1155.40	shear thinning	13331.72	16480	Newtonian	1179% increased	p = 0.04	P = 0.001
	0.1	592.35	596.61		13404.38	15422		2163% increased	p = 0.04	
	1	74.11	73.38		set			set	p = 0.04	
AH Plus	0.05	399.44	626 .86	shear thinning	320.73	438.84	shear thinning	20% reduced	p = 0.35	P = 0.069
	0.1	241.50	381.43		191.11	261.78		21% reduced	p = 0.14	
	1	33.53	47.23		22.75	32.75		32% reduced	p = 0.35	

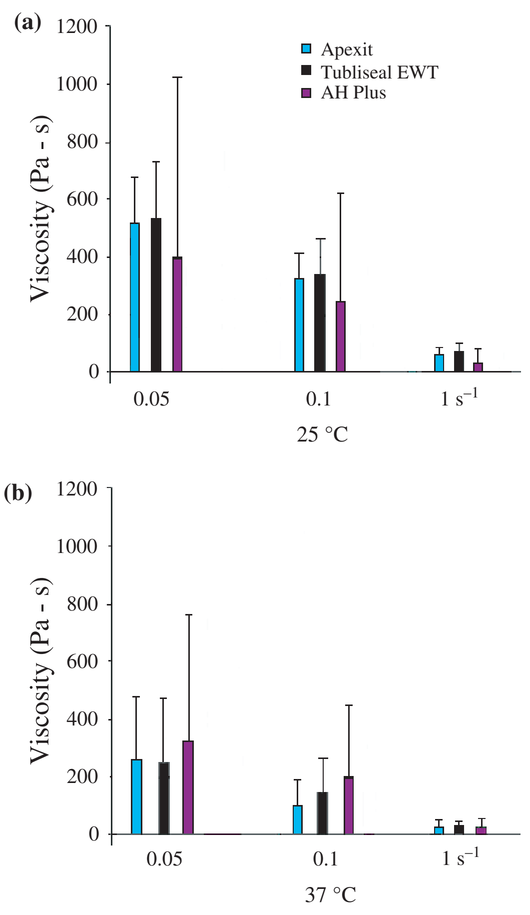

Figure 3 Viscosity of Apexit, Tubliseal EWT and AH Plus at shear rates, 0.05, 0.1 and 1 s^{-1}, and 25 °C (a) and 37 °C (b), standard deviations represented by bars.

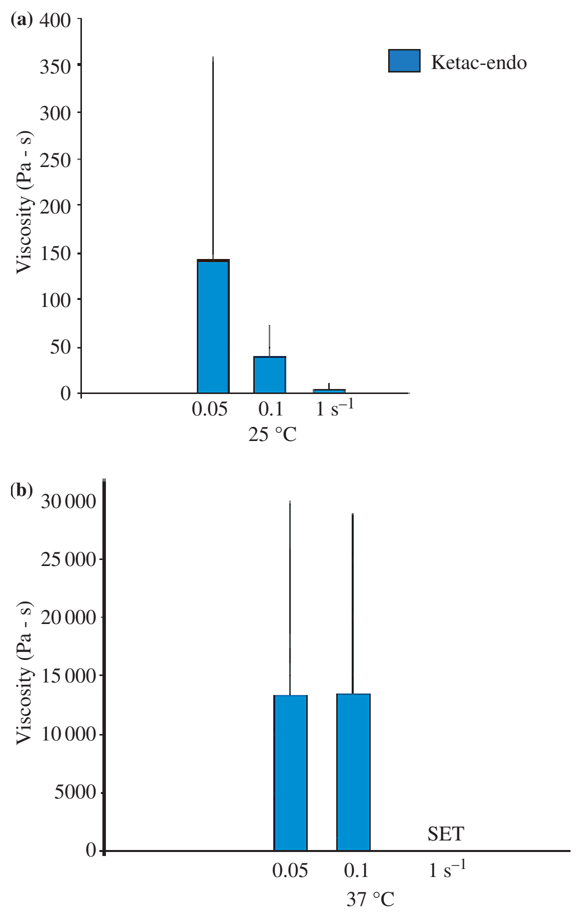

Figure 4 Viscosity of Ketac-endo at shear rates, 0.05, 0.1 and 1 s^{-1}, and 25 °C (a) and 37 °C (b), standard deviations represented by bars.

The ARES rheometer was chosen as it can control the temperature accurately and can perform rheometric characterization over a very wide range of shear rate. The experiments can be run for any length of time as required or until the material sets. Viscosity is an extremely sensitive indicator of chemical change and any discrepancies in this study may be attributed to variation in mixing technique, variations in time from start of mix, in powder particle size, or in humidity even though every attempt was made to control these variables. All materials were measured on a laboratory balance and mixed within manufacturers' stated working time. Excess material was used to avoid the effect of variation in humidity. It can be noted that least variation was registered for the paste : paste systems (Apexit and Tubliseal EWT) and the most for hand-mixed powder : liquid materials (Grossman's 3 : 1 and 2 : 1).

It was estimated from a previous study (Lacey *et al.* 2005) that the shear rate in a clinical situation was likely to be within the range of 0.005–1 s^{-1}. This study has shown that even within this range there is a difference in sealer behaviour. Tubliseal EWT, Apexit and AH Plus all showed shear thinning and reduced viscosity with increased temperature. In contrast, Grossman's sealer and Ketac-endo had increased viscosity. The two powder : liquid ratios of Grossman's showed different behaviour, 2 : 1 being shear thickening at higher temperature, while Grossman's 3 : 1 was shear thinning.

The shear thinning demonstrated by all sealers at 25 °C suggests that increasing shear while inserting the sealer would produce improved flow of the sealer by reducing the viscosity. However, at 37 °C increasing shear strain rate produced an early set and therefore reduced working time in Grossman's 2 : 1, 3 : 1 and

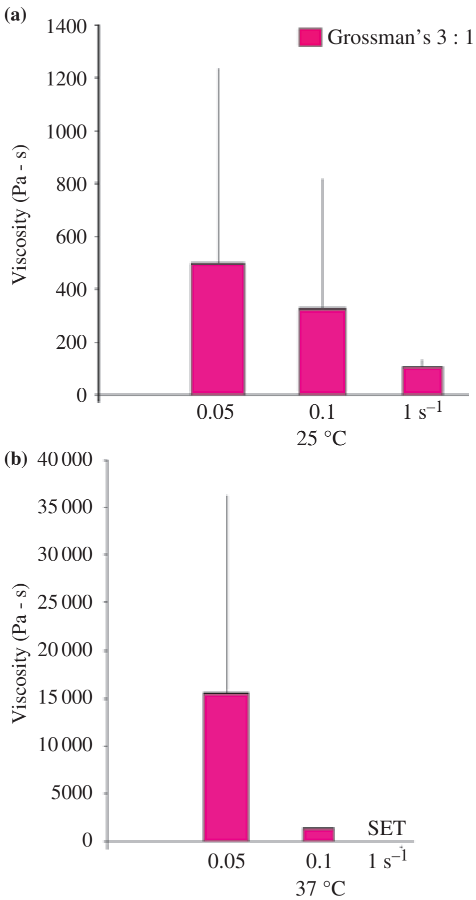

Figure 5 Viscosity of Grossman's powder : liquid ratio 3 : 1 at shear rates, 0.05, 0.1 and 1 s^{-1}, and 25 °C (a) and 37 °C (b), standard deviations represented by bars.

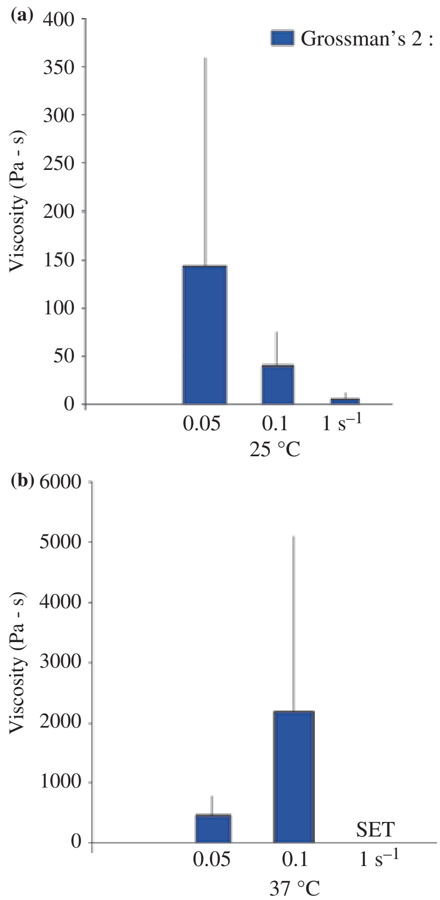

Figure 6 Viscosity of Grossman's powder : liquid ratio 2 : 1 at shear rates, 0.05, 0.1 and 1 s^{-1}, and 25 °C (a) and 37 °C (b), standard deviations represented by bars.

Ketac-endo. At 37 °C, Apexit, Tubliseal EWT and AH Plus would be likely to have improved flow with an increased rate of insertion.

The change in behaviour with increased temperature may not be so relevant in a single-cone technique, but may have more relevance in lateral condensation techniques which take longer to complete. For filling techniques that require a short time, sealer temperature may remain at room temperature (25 °C) and the flow of all sealers may be improved by rapid insertion and placement. Any filling technique that requires a longer time may allow the sealer to reach mouth temperature (37 °C) and there will be differing effects with different sealers. Grossman's and Ketac-Endo will have greatly reduced working time unless inserted very slowly. Apexit, Tubliseal EWT and AH Plus may have better flow if inserted rapidly. As the results for AH Plus have not shown significant difference with increase in temperature, this may be the sealer of choice in filling techniques that take more time. Further research may indicate how quickly each sealer will reach the mouth temperature after insertion and how quickly heat might be transferred through gutta-percha to the sealer.

Most studies that have investigated the flow of sealers have done so either at room temperature or mouth temperature. Two studies, however, compared the viscosities of sealers at room temperature and a reduced temperature. It has been found that reducing the temperature by mixing on a chilled slab or in a water bath (Vermilyea *et al.* 1979, Lorton *et al.* 1980) reduced the viscosity or delayed the increase of viscosity on setting. One study showed reduced flow of sealers with higher temperature (Grossman 1976) but this was in the presence of increased humidity, which would contribute to an earlier set. The results of a previous study using a capillary rheometer at 25 °C demonstrated that rheological behaviour of sealers depends on the geometry of the capillary used (Lacey *et al.* 2005).

This study has shown the effect of increasing the temperature of the setting cements from room temperature to mouth temperature within the manufacturers' recommended setting time and within a likely clinical range of shear.

Conclusion

The flow of root canal sealers was temperature and shear dependent and varied between sealers. On increasing the temperature from 25 °C to 37 °C, Apexit, Tubliseal and AH Plus had reduced viscosity whereas Grossman's (powder : liquid ratios 2 : 1 and 3 : 1) and Ketac-endo had increased viscosity. There was variation in this effect on each sealer depending on the shear rate. The difference in viscosity was significant ($P < 0.05$) in all sealers except AH Plus. These findings apply across a range of shear of 0–50 s^{-1} and within 30 min from start of mixing of the cements. The null hypothesis that there would be no significant difference ($\alpha = 0.05$) in the viscosity of endodontic sealers with increase in temperature from 25 °C to 37 °C was rejected.

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