The rheological properties of seven endodontic sealers were assessed by rotational viscometry as a function of time and rotational speed at a constant temperature. The viscosity in centipoise at 37°C of Roth Cement #811 (A), #801 (B), #601 (C), Kerr Pulp Canal Sealer (D), Tubli-Seal (E), Proco-Sol (F), and Diaket (G) was determined at rotational speeds of 1, 2.5, 5, 10 and 20 revolutions per minute (rpm). Data were recorded initially at two minutes after the start of mixing and continued for a maximum period of 15 minutes. Initial viscosities at 1 rpm were: A, 7,000 cp; B, 9,000 cp; C, 20,000 cp; D, 59,000 cp.

Dental materials, cements, endodontic sealers, rheology and viscosity.
The viscosity-time behavior of the test materials exhibited one of four discrete patterns: (1) low initial viscosity with a gradual change in viscosity over a 15-minute period (materials A and B); (2) moderately low initial viscosity with a rapid increase in viscosity at 7 to 10 minutes after mixing (materials C, D, and E); (3) high initial viscosity with a gradual increase in viscosity over a 15-minute period (material F); and (4) high initial viscosity with an abrupt increase in viscosity over a 1-minute period (material G).

Thixotropy appeared to be an additional feature of the viscosity-time behavior of material E. At increased rotational speeds material A was Newtonian whereas the behavior of the other materials was characterized by pseudoplasticity. At 20 rpm the initial viscosities were: A, 7,700 cp; B, 5,000 cp; C, 10,000 cp; D, 18,000 cp; E, 18,000 cp; F, 36,000 cp; and G, 65,000 cp. The data make possible the selection of specific materials to meet the clinical needs of both the experienced and inexperienced operator.
THE RHEOLOGICAL PROPERTIES OF ENDODONTIC SEALERS

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Commercial materials and equipment are identified in this report to specify the experimental procedure. Such identification does not imply official recommendation or endorsement or that the equipment and materials are necessarily the best available for the purpose.

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Previous studies have demonstrated the importance of sealers in endodontic treatment.\textsuperscript{1-3} It would appear that the actual sealing of the properly instrumented canal is accomplished by the sealant itself. The ability of a paste-sealant to fill small accessory canals which do not allow entrance of solid core materials is dependent upon flow characteristics. These characteristics, in turn, are governed largely by rheological phenomena. Unfortunately, sufficient information relevant to the flow properties of endodontic sealers is not available.

The present investigation was conducted to determine the apparent rheological (viscous) properties of several endodontic sealers as a function of time and shear rate at a constant temperature.

Materials and Methods

Seven endodontic sealers (Diaket,\textsuperscript{*} Kerr Pulp Canal Sealer,\textsuperscript{+} Tubli-Seal,\textsuperscript{+} Proco-Sol\textsuperscript{#} and Roth Cement Types 601\textsuperscript{\textregistered}, 801\textsuperscript{\textregistered} and 811\textsuperscript{\textregistered}) were obtained commercially. Each material was prepared in accordance with its manufacturer's recommendations to yield volumes of about 1 cm\textsuperscript{3}. Mixing was accomplished at 23±2\textdegree C and 50±10\% relative humidity. Each sample of the mixed material was placed in a disposable vial and suspended in a water bath at 37±0.1\textdegree C throughout the time of viscosity measurement.

\textsuperscript{*} Premier Dental Products Co., Norristown, PA 19401.
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\textsuperscript{\textregistered} Roth Chicago, IL 60610.
Viscosities of the test materials were determined with a rotational viscometer at rates of 1, 2.5, 5, 10 and 20 revolutions per minute (rpm). Five trials were made with each material at each rotational speed. Data were obtained as viscosities in centipoise (cp) by calibration of the viscometer's t-bar spindle with a Newtonian Standard. Initial viscosities of all of the materials were recorded at two minutes after the termination of mixing. Viscosities were measured continuously for a period of 15 minutes following the end of mixing or until the viscosity of the mix exceeded the range of the measuring instrument.

Results

The changes in the viscosity of each material with time at a rotational speed of 1 revolution per minute are shown in Figures 1 and 2. Initial viscosities ranged from 7,000 cp for Roth Cement Type 811 to 678,000 cp for Diaket. The viscosity of six of the materials increased with time. Roth Cement Type 801, Roth Cement Type 811 and Proco-Sol showed a gradual increase in viscosity during the 15-minute time of measurement whereas Roth Cement Type 601 and Kerr Pulp Canal Sealer exhibited an abrupt increase in viscosity after 10 minutes and 12 minutes, respectively. The viscosity of Tubli-Seal decreased from 123,000 cp to 106,000 cp at five minutes after the cessation of mixing. Subsequently, however, the viscosity of Tubli-Seal increased sharply to more than 160,000 cp at 9 minutes after mixing.


¶ Viscosity Standard 100,000, Brookfield Engineering Corp., Stoughton, MA 02074.
Diaket demonstrated a rapid increase in viscosity over the entire time of measurement.

The change in viscosity with increased rotational speed at 2 minutes after mixing is shown in Figures 3 and 4. The viscosity of six materials decreased markedly with increased rotational speed. The viscosity values at 20 revolutions per minute were 90 percent (Diaket) to 42 percent (Roth Cement Type 801) less than those measured at 1 revolution per minute. The viscosity of one material (Roth Cement Type 811) at 20 rpm was not significantly different from that measured at 1 rpm.

Discussion

The viscosity and flow properties of endodontic sealers are of importance in their clinical application. Materials with a viscosity similar to that of water (1 cp) would be difficult to control during mixing and intraradicular placement. Conversely, extremely viscous (viscosity greater than one million cp) materials would be difficult if not impossible to mix and to deposit onto the walls of the prepared canal.

Viscosity of the sealers is affected by many factors. The differences in the initial viscosities of the proprietary sealers may be attributed to variations in particle size, powder-liquid or base-catalyst ratios or rates of setting of the mixed materials. The clinical significance of these differences, however, is unclear. Optimal viscosities required for the obturation of root canal systems have not been established.
The initial decrease in apparent viscosity with time at a constant rotational speed exhibited by Tubli-Seal probably reflects the thixotropic nature of this material. Thixotropy (the decrease in viscosity with time at a constant rate of shear) reduces the viscosity of the material during mixing and upon its spin-deposition into the root canal system.

Shearing of an endodontic sealer occurs during its mixing and placement into the root canal system and also upon the subsequent condensation of a solid filling material (gutta percha). The pseudoplastic or shear thinning behavior exhibited by six of the test materials suggests that rapid spatulation, high rates of rotation of sealer-laden endodontic instruments and high condensation pressures would reduce the viscosities of these materials. Although rates of shear employed in clinical practice are unknown, it is likely that the apparent effect of shear on the viscosity of sealers would facilitate their manipulation and introduction into small accessory canals.

Analysis of the data suggests that the test materials, with respect to time, exhibit one of four patterns of viscosity change:

1. Low initial viscosity with a gradual change in viscosity over a 15-minute period (Roth Cement Type 801 and Type 811);
2. Moderately low initial viscosity with a rapid increase in viscosity at 7 to 10 minutes after mixing (Kerr Pulp Canal Sealer, Tubli-Seal and Roth Cement Type 601);
3. High initial viscosity with a gradual change in viscosity over a 15-minute period (Proco-Sol) and
4. High initial viscosity with an abrupt increase in viscosity over a 1 to 2-minute period (Diaket).
Viscosity-time behavior patterns provide criteria essential to the rational selection of endodontic sealants. Materials exhibiting small increases in viscosity over reasonably long periods of time would appear to be of benefit to the inexperienced operator when multiple canals are to be obturated or when minor adjustment of solid cores (gutta percha or silver cones) may be required. On the other hand, materials that exhibit abrupt increases in viscosity over relatively short periods of time may be preferred by the more experienced clinician.

Conclusions

Wide variation in the initial viscosities of seven endodontic sealers was demonstrated by rotational viscometry. All of the materials exhibited increased viscosity with time. However, the viscosity of one material increased at a markedly faster rate.

The behavior patterns of six test materials were characterized by a decrease in initial viscosity with increased rotational speed. The flow of such sealants into accessory canals may be enhanced by their placement into the root canal system at high rates of shear.

The data facilitate the selection of specific materials to meet the clinical needs of both the experienced and inexperienced operator.
References


Legends for Figures

Figure 1. Changes in viscosity with time: (A) Roth Cement Type 811; (B) Roth Cement Type 801; (C) Roth Cement Type 601; (D) Kerr Pulp Canal Sealer and (E) Tubli-Seal.

Figure 2. Changes in viscosity with time: (F) Proco-Sol and (G) Diaket.

Figure 3. Changes in viscosity with rotational speed: (A) Roth Cement Type 811; (B) Roth Cement Type 801; (C) Roth Cement Type 601 and (D) Kerr Pulp Canal Sealer.

Figure 4. Changes in viscosity with rotational speed: (E) Tubli-Seal; (F) Proco-Sol and (G) Diaket.
Fig. 1