

The Influence of Temperature on Rheological Properties of Three Root Canal Sealers

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Abstract: Purpose was to determine the viscoelastic properties of three root canal sealers as a function of temperature, time and frequency using dynamic oscillatory measurements. Methods: Measurements were performed on the dynamic oscillatory rheometer set to temperatures: 25°C, 35°C, 40°C and 65°C. Stress sweep and frequency sweep tests were used in order to determine storage moduli G' , loss moduli G'' and complex viscosity η^* . Results Higher values of storage compared to loss moduli indicate the more pronounced solid-like behavior of tested materials. EndoREZ showed the highest values of G' and G'' moduli at all temperatures. With temperature increase the G' and G'' moduli of Sealapex and EndoREZ increased while the moduli of AH Plus decreased. With frequency increase all materials showed viscosity decrease exhibiting non-Newtonian, shear-thinning behavior. With temperature increase AH Plus demonstrated viscosity decrease, while the viscosity of Sealapex and EndoREZ increased. Clinical significance: All tested materials were temperature, time and frequency dependent and this dependency varied between them. The obtained results may be used to predict the rheological behavior of root canal sealers in different temperature conditions, thus helping as to estimate optimal handling characteristics for specific clinical applications.

Keywords: rheological properties, root canal sealer, dental materials, handling characteristics

1. Introduction

Root canal sealers (RCS) are materials used in endodontic therapy to fill the spaces among guttapercha cones or other solid core material and dentinal wall, but also the intracanal irregularities, apical ramifications, lateral canals and dentinal tubules. In order to provide a three-dimensional canal seal, various obturation techniques have been developed: lateral compaction of guttapercha, cone-fit, warm vertical compaction, warm lateral compaction, continuous wave, carrier-based obturation techniques. While some studies have found that none of the existing obturation techniques cannot achieve superior canal filling [1-3], others suggest that thermoplasticized guttapercha obturation techniques produce better filling of lateral canals and provide more favorable outcomes of root canal treatments [4,5]. When it comes to warm guttapercha obturation techniques, there are studies have found significantly lower temperature of guttapercha measured in root canal comparing to temperature shown at the display of heat source device (160-200°C); these temperatures ranges from 61 to 70°C [6-9]. Although the heated guttapercha flows better into narrow, peripheral parts of the root canal system, the influence of temperature on sealer properties has been questioned. In previous study was found that the amine groups of the AH Plus (epoxy resin based sealer) were affected by the heat producing lower compressive strength, while the chemical compositions of calcium silicate and zinc oxide-eugenol based RCS were not affected [7]. Furthermore, the setting time and the film thickness of the resin based RCS seemed to be affected by heat [10].

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Although manufacturers state that sealers are suitable for both heated and cold guttapercha obturation techniques, it is indisputable that the properties of the sealer change under high temperature [7,8,10,11].

Rheological properties of material affect its handling characteristics and clinical behavior [12-15]. The viscosity of the RCS is clinically important because it determines the way material flows into root canal, thus affecting the clinical performance of the material. These properties greatly affect the restorative procedure, treatment time and the clinical outcome. Many previous studies have determined the sealer flowability using a simple press method, regulated by ISO standard (6876/2001) and ANSI/ADA specifications (No. 57), based on the diameter of sealer pressed between two glass slabs loaded by controlled weight for a certain amount of time. The methods are relatively simple to perform, but obtained information are very limited [12,13,16-21]. RCS have viscoelastic properties between pure elastic solid and viscous liquid, having more viscous-like or solid-like behavior. By using stress controlled rheometer and dynamic oscillatory measurements it is possible to quantify the both components. The elastic (storage) modulus G' measures the energy stored inside the material, while the viscous (loss) modulus G'' measures the dissipated energy [12,21,22]. Limited data are available about viscoelastic properties of RCS when they are exposed to high temperature and their suitability for warm guttapercha obturation techniques.

The aim of the present study was to determine the viscoelastic properties of three root canal sealers as a function of temperature, time and frequency using dynamic oscillatory measurements.

2. Materials and methods

2.1. Materials

Three different commercially available RCS were tested in this study. The chemical compositions and the manufacturer's information are given in Table 1.

The materials were mixed and prepared according to the manufacturer's instructions: AH Plus and EndoREZ components were packed in dual syringes and mixed automatically in the 1:1 ratio; Sealapex was mixed manually, using a precise scale by which the equal amounts of both sealer components were measured and mixed.

Table 1. The Root canal sealers used in the study

Material – type	Chemical composition	Manufacturer
AH Plus-Epoxy resin based	Paste A: epoxy resin, calcium tungstate, zirconium oxide, aerosil, iron oxide pigments Paste B: amines, calcium tungstate, zirconium oxide, aerosil, silicone oil	Dentsply, De Trey, Konstanz, Germany
Sealapex – Calcium hydroxide containing polymeric resin	Base:: N-ethyl toluene sulfonamide resin, silica, zinc oxide, calcium oxide Catalyst: Isobutyl salicylate resin, polymethyl methacrylate, methyl salicylate, silica, bismuth trioxide, titanium dioxide pigment	Kerr, Salerno, Italy
EndoREZ – Methacrylate resin based	Base: Diurethane dimethacrylate, benzoyl peroxide Catalyst: 2,2'-(p-Tolylimino)diethanol, triethylene glycol dimethacrylate	Ultradent, South Jordan, Utah, USA

2.2. Rheological tests

The test methods used in the study are previously described in [14,15,23]. Dynamic oscillatory measurements were performed using a HAAKE Mars Rheometer (Thermo Fisher Scientific, Karlsruhe, Germany) and parallel plates geometry (diameter of 20mm) at 25°C (room temperature), 35°C (intracanal temperature), 40 and 65°C. Freshly prepared sealers were delivered on the lower plate, preheated to the selected temperature; upper plate was moved downwards until 1 mm gap between the plates was achieved. The excess material was discarded, Teflon cover was used as a light protection, and entire procedure was carried out in darkened room. In order to achieve the stress relaxation in the material, the measurements were started two minutes after the compression of the sample. The *amplitude*

sweep tests were performed in the range from 0.01 to 50 Pa in order to determine the linear viscoelastic region (LVR)- the interval of shear stresses that give constant values of G' and G'' moduli at constant frequency (1 Hz); the selected values of shear stress were kept constant in further testing. *Frequency sweep* tests were performed in the range from 0.1 Hz to 10 Hz while the storage shear moduli G' , viscous shear moduli G'' and complex viscosity η^* were recorded. Three measurements were performed for each group; three consecutive measurements were performed on every sample.

Statistical analysis was performed using the One-way ANOVA test and post-hoc Tukey HSD, with significance level $p < 0.05$.

3. Results and discussions

The results of the *frequency sweep* tests for all tested materials are presented on Figure 1-3, in logarithmic scales. Higher values of storage G' compared to loss moduli G'' indicate the more pronounced solid-like behavior of each material. For almost all groups both moduli increased with frequency, except G'' of Sealapex at 65°C that was approximately constant. In the range of applied frequencies and shear stresses EndoREZ showed the highest values of both moduli at all measured temperatures. The temperature increase influenced the G' and G'' moduli in a different manner: moduli of Sealapex and EndoREZ increased while the moduli of AH Plus decreased.

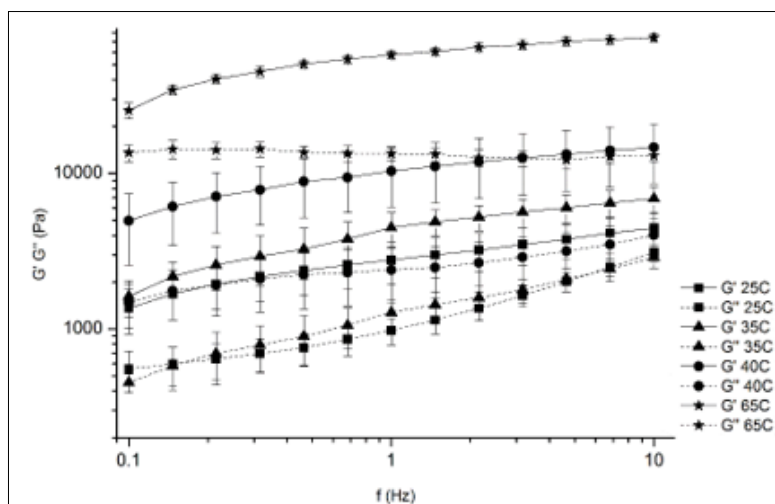


Figure 1. Frequency sweep test: the storage G' and loss G'' moduli versus frequency for AH Plus at 25°C, 35°C, 40°C and 65°C

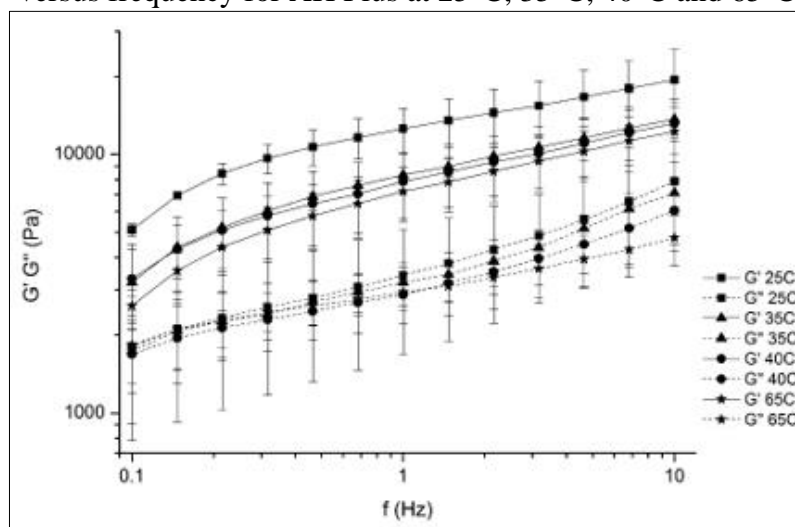


Figure 2. Frequency sweep test: the storage G' and loss G'' moduli versus frequency for Sealapex at 25°C, 35°C, 40°C and 65°C

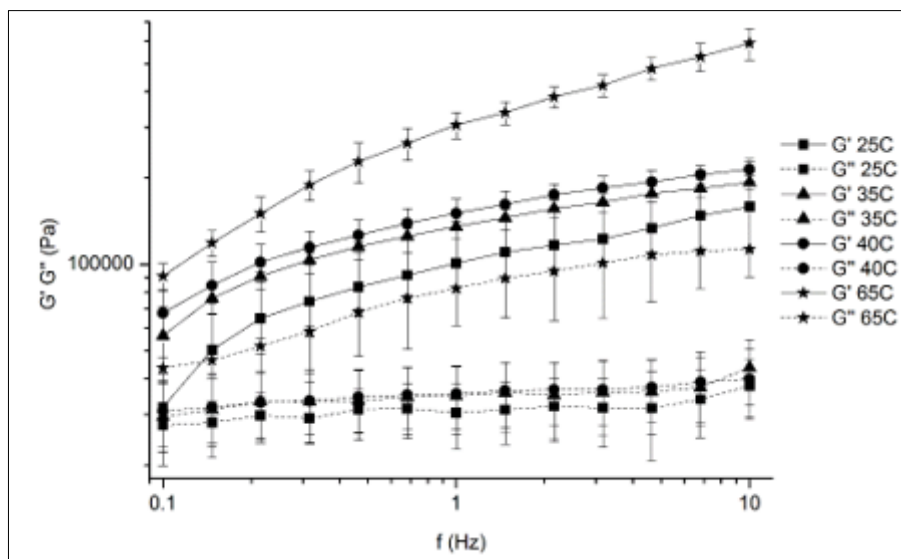


Figure 3. Frequency sweep test: the storage G' and loss G'' moduli versus frequency for EndoREZ at 25°C, 35°C, 40°C and 65°C

The complex viscosity, $\eta^* = G^*/\omega$, where $G^* = T_A/\gamma_A$ with T_A being a shear stress amplitude and γ_A being a strain amplitude, versus frequency for each material are presented on Figure 4-6, in logarithmic scales. Complex viscosity decreased as frequency increased for all tested sealers. If it is assumed that the Cox-Merz rule is valid, it can be concluded that all tested materials exhibit the non-Newtonian, pseudoplastic behavior [21, 24]. With temperature increase AH Plus showed viscosity decrease, while the Sealapex and EndoREZ behaved differently and showed increase in viscosity.

By individual material analysis, it can be noted that only the complex viscosities of Sealapex and EndoREZ at 65°C were significantly higher ($p < 0.05$) than the values at low temperatures; differences between values at 25°C, 35°C and 40°C were not statistically significant. By comparative materials analysis at the same temperature, it can be noted that EndoREZ had significantly higher complex viscosity values than AH Plus and Sealapex at all measured temperatures ($p < 0.05$).

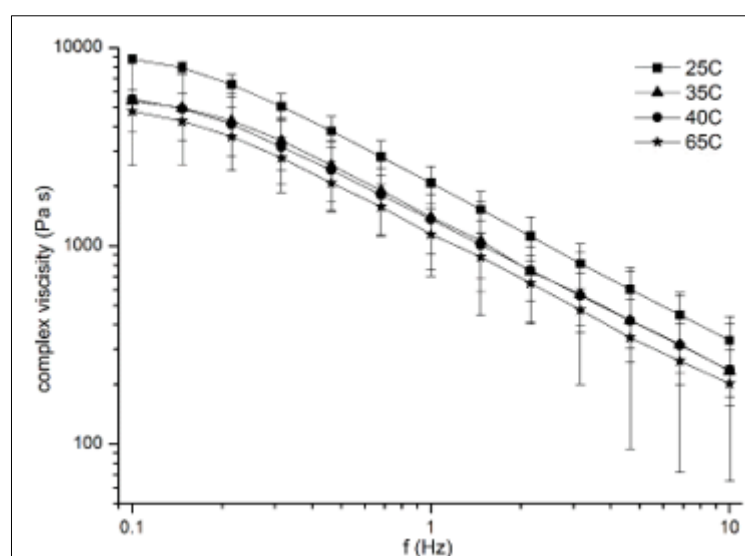


Figure 4. Frequency sweep test: complex viscosity versus frequency for AH Plus at 25°C, 35°C, 40°C and 65°C

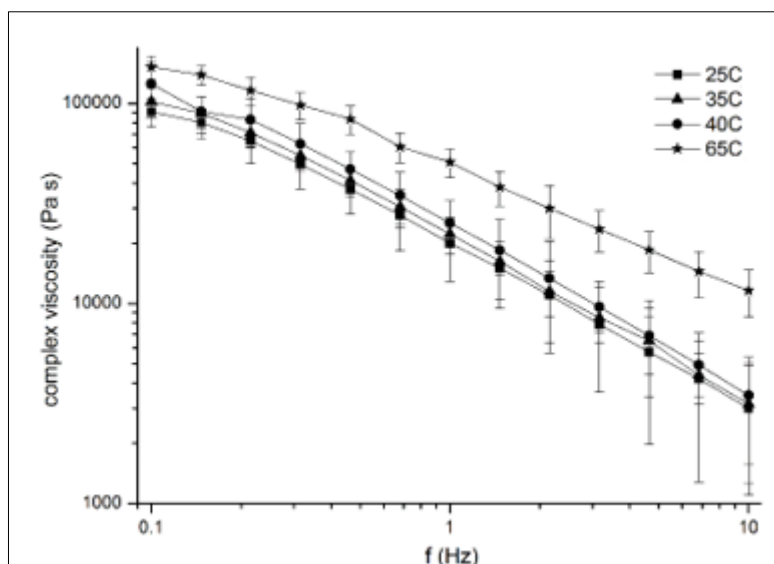


Figure 5. Frequency sweep test: complex viscosity versus frequency for Sealapex at 25°C, 35°C, 40°C and 65°C

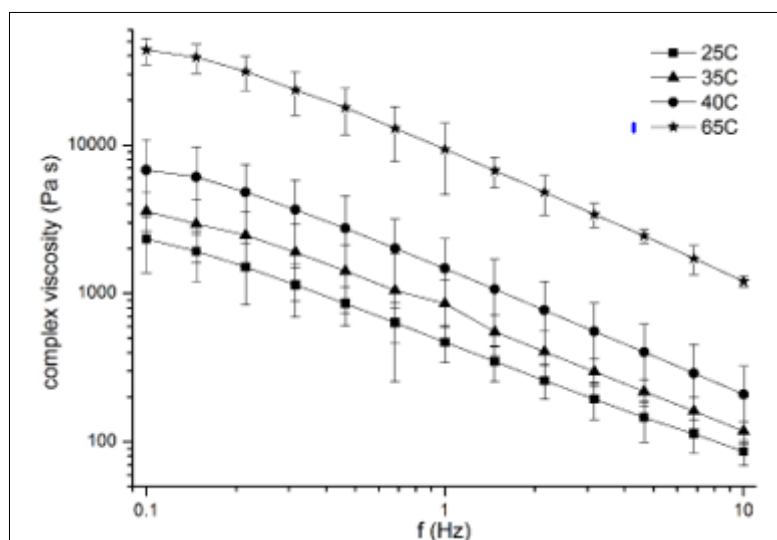


Figure 6. Frequency sweep test: complex viscosity versus frequency for EndoREZ at 25°C, 35°C, 40°C and 65°C

At 25°C and 35°C AH Plus showed higher complex viscosity than Sealapex; at 40°C the values of these two sealers were approximately the same; at 65°C Sealapex showed higher complex viscosity. Differences in complex viscosity among AH Plus and Sealapex were not statistically significant.

Upon three consecutive measurements on each sample it has been noted an increase of the complex viscosity for all tested materials at each temperature (rheopexy behavior), except AH Plus at 65°C where viscosity decreased (thixotropy behavior). EndoREZ showed the greatest changes over time, particularly at 65°C, while AH Plus and Sealapex showed a similar increase in viscosity over time at all set temperatures.

All tested materials were two-components, paste / paste, from the same batch, prepared according to the manufacturer's instructions. A wide range of standard deviations of G' , G'' and η^* was noted in *frequency sweep* tests. Lacey et al. [12] also noticed the wide standard deviations when it comes to viscosity measurements of RCS. On the other hand, in the studies of Petrovic *et al.* [14,15] where one-component dental materials were tested (composite materials), using the same experimental method, the reproducibility of *frequency sweep* test was satisfactory. Rheological properties of two-component



materials are highly sensitive indicator of any change such as: variations in the amount of each component, mixing technique, mixing time, measurements time, temperature and humidity. In the present study auto-mix syringes were used for the mixing of AH Plus and EndoREZ; precision scale was used for ensuring the extrusion of the equal amounts of both components of Sealapex; measurement time was uniform and strictly controlled; teflon cover was used as a protection from light and humidity. Baldi et al. [16] demonstrated the variations of physicochemical properties of epoxy-based RCS (AH Plus) depending on the segment of the tubes from which the components were extruded and mixed. Namely, sealer flowability (ANSI/ADA specification No. 57) had the highest value if the components were mixed from the beginning parts of tubes and when sealer was mixed from the lower parts of the same tubes, the flowability decreased. Non-uniform consistency of components in the tubes can also explain the wide standard deviations when the flow characteristics and viscoelastic properties of RCS are measured. Stress controlled rheometer and dynamic oscillatory shear measurements are highly precise and able to detect even minimal structural changes in the material.

The temperature of 65°C was used in the present study as the average temperature of guttapercha achieved in the canal when warm vertical compaction is used, according to literature data [6-9]. It should be kept in mind that although the display of heat source device shows temperatures up to 200°C, the guttapercha in the canal reaches a significantly lower temperature [6-9]. The experimental method in the present study provided a high level of condition standardization, which is very difficult to achieve *in vivo*. In *in vivo* conditions the viscoelastic properties of sealer may be altered due to manipulation of heated gutta-percha and various morphology of root canal system [25].

All tested materials behaved like non-Newtonian, shear-thinning fluids, i.e. as frequency increased the viscosity decreased, at all temperatures. These results are in line with the findings from other similar studies [12,13,21,24-26]. It indicates that an increase of the shear rate while handling the sealer would produce lower viscosity, i.e. improved flow properties. High flowability will produce better penetration into intracanal irregularities and around the core material, but also the higher risk of apical extrusion. According to test results, under high temperature the complex viscosity of AH Plus decreased while of the Sealapex and EndoREZ increased (these properties were more pronounced for EndoREZ). Regarding AH Plus, the results are in agreement with the findings of Lacey et al. [12] and Khedmat et al. [27]; AH Plus exhibits like simple polymeric suspensions [28]. As described in previous study, amine groups of the AH Plus seemed to be affected by temperature increase [7]. The high viscosity may impede the injection of the RCS into the root canal, potentially resulting in formation of gaps and voids that may compromise the treatment outcome. Low-viscosity materials exhibit greater adaptability along the interface between the gutta-percha and dentinal wall. By knowing viscosity changes under high temperature, material can be handled properly. According to the results from this study, if more flowable material is required, for example in the cases where apical delta or narrow root canal exist, AH Plus will better flow in combination with thermoplasticized gutta-percha obturation techniques, while Sealapex and EndoREZ should be combined with cold gutta-percha techniques. Otherwise, if a root has wide and open apex and high flowability is not required, AH Plus should be combined with cold gutta-percha obturation techniques, while Sealapex and EndoREZ is better to combine with thermoplasticized gutta-percha techniques. Further clinical research is necessary for determining the optimal viscosity of RCS.

The *frequency sweep* test was repeated three times on every sample in order to investigate the viscoelastic properties of material in the course of time. Repeating the *frequency sweep* test in succession is possible due to its non-destructive characteristics, i.e. applied stress is too small so the material properties are not affected. The complex viscosity of all tested sealers increased over time, except AH Plus at 65°C where viscosity decreased. Increase in viscosity is in accordance with the setting time reactions of materials; this is defined as rheopexy behavior [29]. Namely, EndoREZ showed the highest increase of complex viscosity (particularly at 65°C) with time which was expected due to its short working (12-15 min) and setting time (20-30 min), as stated by the manufacturer. EndoREZ is a urethane dimethacrylate-based RCS with hydrophilic characteristics, so it differs from other sealers in that respect. Clinically, EndoREZ should be handled quickly, especially if it is combined with thermo-



plasticized gutta-percha obturation techniques. EndoREZ is not material of choice for time-consuming obturation techniques; it is suitable for less time-consuming techniques such as single-cone technique. Contrary, AH Plus is two-component resin based sealer with long working and setting time; the polymerization reaction of amines and epoxy resin is slow process [27]. Clinically, AH Plus and Sealapex have not shown great changes over time and may be used in time-consuming obturation techniques.

The present study may contribute to an improved understanding of the rheological behavior of RCS. The obtained results may be used to predict the rheological behavior of root canal sealers in different clinical and temperature conditions, thus helping as to estimate optimal handling characteristics for specific clinical situations.

4. Conclusions

Within the limitations of this study it can be concluded:

All tested RCS showed temperature, time and frequency - dependent changes.

Different rheological behavior can be noted among tested materials, particularly when heat was applied, although they are all used for the same clinical procedure. This implies that further investigations are necessary in order to determine the influence of complex viscosity, storage and viscous moduli on the clinical performance of RCS.

Further clinical research is required to determine the optimal viscosity of RCS.

Acknowledgments: This research was supported by the Serbian Ministry of Education and Science [grant number 174005 entitled “Viscoelasticity of fractional type and optimization of shape in rod theory”].

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Manuscript received: 9.02.2022