**Original Article**

# A Comparison of Viscoelastic Properties of Three Root Canal Sealers

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**Abstract**

**Objective:** Handling of endodontic sealers is greatly dependent on their elasticity and flow ability. We compared the viscoelastic properties of three root canal sealers.

**Materials and Methods:** AH Plus (Dentsply, De Trey, Konstanz, Germany), Endofill (Dentsply Hero, Petrópolis, Rio de Janeiro, Brazil) and AH26 (Dentsply, De Trey, Konstanz, Germany) were mixed according to the manufacturers' instructions. The resulted pastes were placed on the plate of a rheometer (MCR 300, Anton-Paar, Graz, Austria). The experiments were performed at 25˚C and 37˚C. Viscoelastic properties of the sealers including loss modulus (G"), storage modulus (G´) and complex viscosity (η*) were studied using dynamic oscillatory shear tests. The shear module versus frequency (from 0.01 to 100 $S^{-1}$) curves were gained using frequency deformation sweep test. Three samples of each material were examined at each temperature. The mean of these three measurements were recorded.

**Results:** The storage modulus of AH plus was higher than its loss modulus at two temperatures. Endofill exhibited a crossover region in which the storage modulus crosses the loss modulus in both temperatures. At 25ºC the loss modulus of AH26 was higher than the storage modulus (G">G′). In contrast, at 37ºC G′was greater than G″ (G′>G″). Both shear modules of AH Plus and Endofill decreased as the temperature raised from 25ºC to 37ºC. On the contrary, the loss modulus and storage modulus of AH26 increased at 37ºC.

**Conclusion:** In both test temperatures, AH Plus behaved like viscoelastic solids and Endofill exhibited a gel-like viscoelastic behavior. AH26 at 25ºC behaved like liquids, while at 37ºC it was an elastic solid-like material.

**Key Words:** Endodontics; Rheology; Sealer

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## INTRODUCTION

Complete obturation of the root canal system plays a chief role in successful endodontic therapy. The use of semisolid points with a paste “sealer” or “cement” is the most common root canal filling system [1,2]. In general, paste-type dental materials are introduced as viscoelastic materials [3,4].

Viscoelastic materials are located between elastic materials, such as metals, and viscous materials, such as oils. Viscoelastic behavior of an endodontic sealer is an indicator of its flow ability and elasticity.

The total values of viscosity and elasticity of a sealer and the ratio of viscosity to elasticity are very important factors that determine the response aspects of the material to external forces [5]. Handling of endodontic sealers is greatly dependent on their elasticity and flow ability [5]. Previous studies have exhibited that the temperature, humidity, shear rate and powder to liquid ratio can affect the viscosity of endodontic sealers [1, 6-10]. In order to measure the viscoelastic behavior of materials, steady shear tests, stress relaxation tests, creep tests and dynamic tests especially dynamic oscillatory shear tests are frequently used [3, 5, 11]. AH26 and AH Plus are two resin-based sealers most commonly used in endodontic treatment [6].

Endofill is a eugenol-based low cost commercially available sealer [12].

To the best of our knowledge, the viscoelastic behavior of endodontic sealers, which is defined as storage modulus (G´), loss modulus (G"), and complex viscosity (η*) versus frequency [5], has not been evaluated in previous investigations. G′ is the elastic modulus, which is a measure of stored energy without phase difference between stress and strain and represents the elastic component of the material. G″ represents the viscosity of the materials and is a measure of the energy lost as heat. The ratio of G″ to G′ (G″/G′) represents the ratio of the viscous part to the elastic part (energy loss/energy stored) of the materials. Dynamic shear viscosity (η*) or complex viscosity reflects the response of the microstructure of the materials to shearing motion of extremely low strength that is identified in the linear dynamic tests [5].

The purpose of this in vitro study was to compare the viscoelastic properties of three root canal sealers through dynamic oscillatory shear tests and to investigate the effect of changes in temperature on their viscoelastic behavior. This is a different outlook of our previous study [10].

## MATERIALS AND METHODS

The test sealers were AH Plus (Dentsply, De Trey, Konstanz, Germany), Endofill (Dentsply Hero, Petrópolis, Rio de Janeiro, Brazil) and AH26 (Dentsply, De Trey, Konstanz, Germany).

The materials were freshly mixed according to the manufacturers' instructions. The resulted pastes were placed on the plate of a cone-plate rheometer (MCR 300, Anton-Paar, Graz, Austria) for each experiment. In order to make a proper analysis and comparison, the samples were weighed precisely with a digital balance (CP124S, Sartorius; Germany) of 0.1mg readability. The experiments were performed at 25˚C (room temperature) and 37˚C (mouth temperature) for each sealer. The effect of changes in relative humidity was minimized by ensuring that there was an extra sealer within the outer rim of the cone. Viscoelastic properties of the sealers including loss modulus (G"), storage modulus (G´) and complex viscosity (η*) were studied using dynamic oscillatory shear tests.

For oscillatory shear measurements, at first the frequency was fixed at 10 s-1 and the shear module of the samples as a function of strain amplitude was measured.

This led to a linear viscoelastic region where the shear module was independent of the amplitude of the applied strain at different given frequencies.

Once the linear region was established, measurements were then taken as a function of frequency at certain amplitudes. The shear module versus frequency (from 0.01 to 100 S-1) curves were gained using frequency deformation sweep test. Three samples of each material were examined at each temperature [5,7,10]. The mean of these three measurements were recorded.

**RESULT**

The results are shown in fig 1 and 2. In this study, deviation from linear viscoelasticity occurred at strains less than 0.5%. Therefore, the frequency sweep measurements were carried out at 0.1% of deformation for all sealers. The mean variations of the shear module versus frequency for three sealers were recorded. Two shear modules including storage modulus (G´) and loss modulus (G") of each sealer were compared separately at each temperature (Fig 1A-C). The storage modulus of AH Plus was higher than its loss modulus at two temperatures (Fig 1A). Endofill exhibited a crossover region while subjected to increasing frequency in which the storage modulus crossed the loss modulus in both temperatures (Fig 1B).

At 25ºC the loss modulus of AH26 was higher than the storage modulus (G"> G′). In contrast, at 37ºC G′was greater than G″ (G′ > G″). Furthermore, at 37ºC both modules were nearly constant (independent of frequency) over the entire experimental frequency range (Fig 1 C). Both shear modules of AH Plus and Endofill decreased as the temperature raised from 25ºC to 37ºC. On the contrary, the loss modulus and storage modulus of AH26 increased at 37ºC. At 37ºC, AH Plus and AH26 at high frequencies exhibited a plateau in which both of the shear modules were nearly constant despite the increasing frequency.

As shown in fig 2 the values of the complex viscosity $|\eta^*|$ of three sealers were greater than the steady shear viscosity (η).

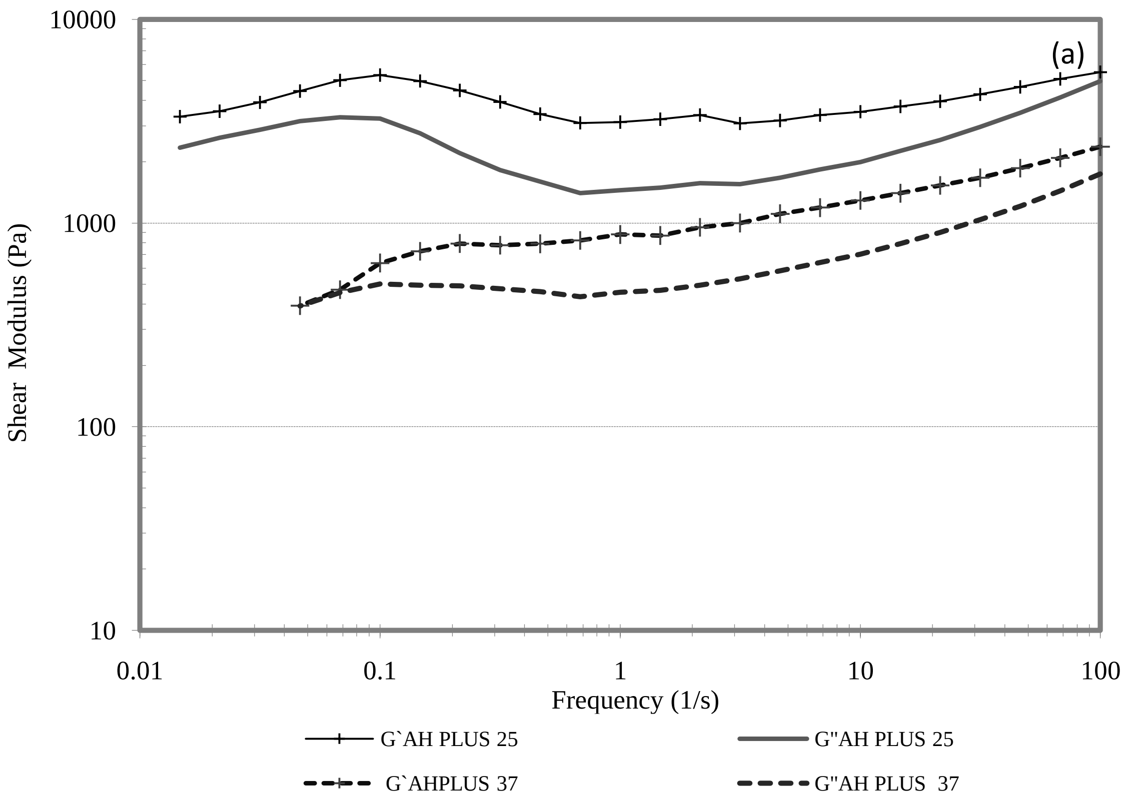


**Fig1 A.** The mean variations of storage modulus (G´) and loss modulus (G") versus frequency of AH plus at two temperatures

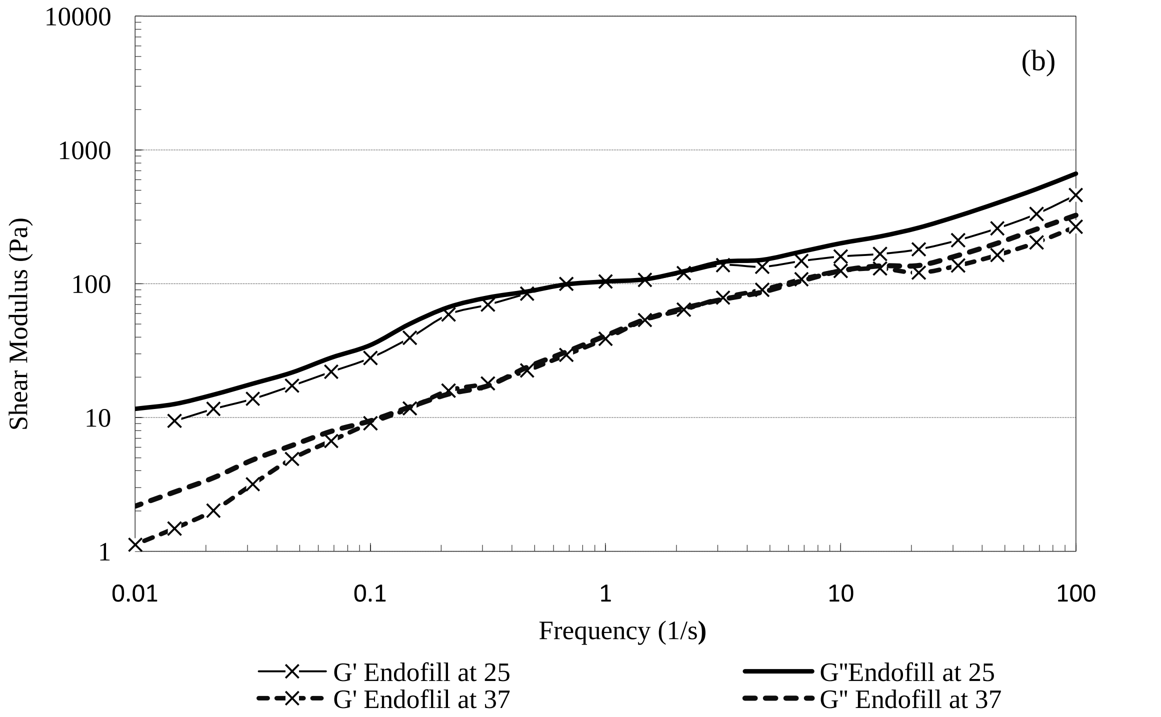


**Fig1 B.** The mean variations of storage modulus (G´) and loss modulus (G") versus frequency of Endofill at two temperatures

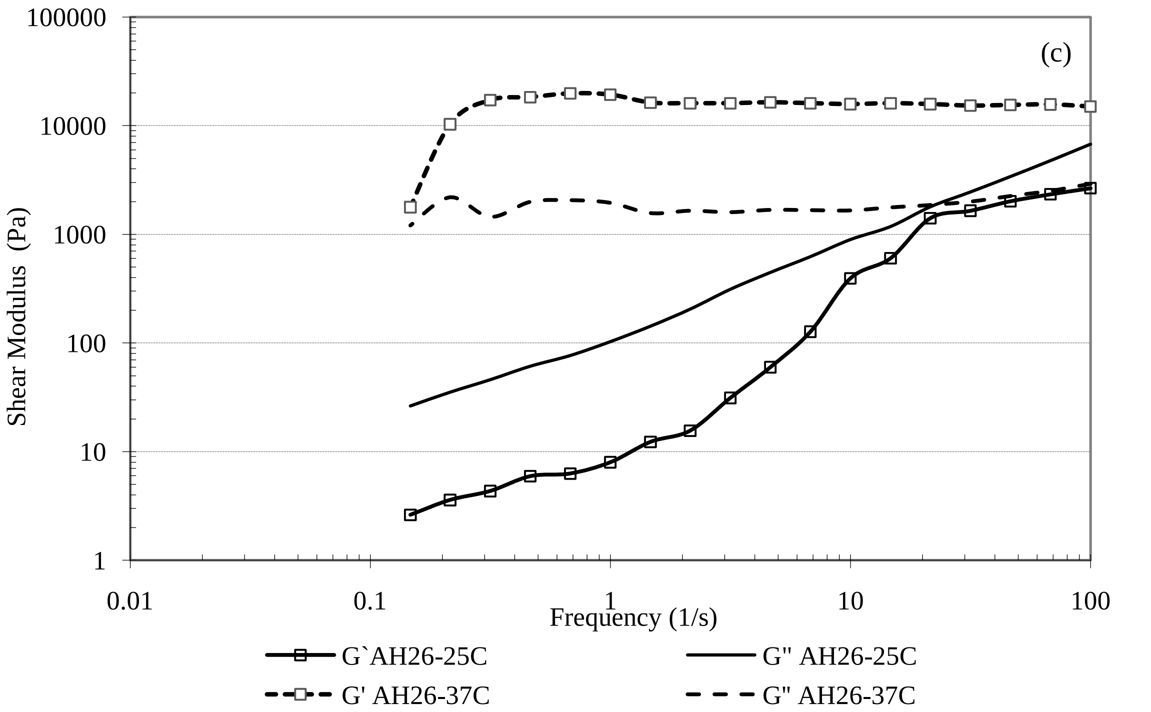


**Fig1 C.** The mean variations of storage modulus (G´) and loss modulus (G") versus frequency of AH 26 at two temperatures

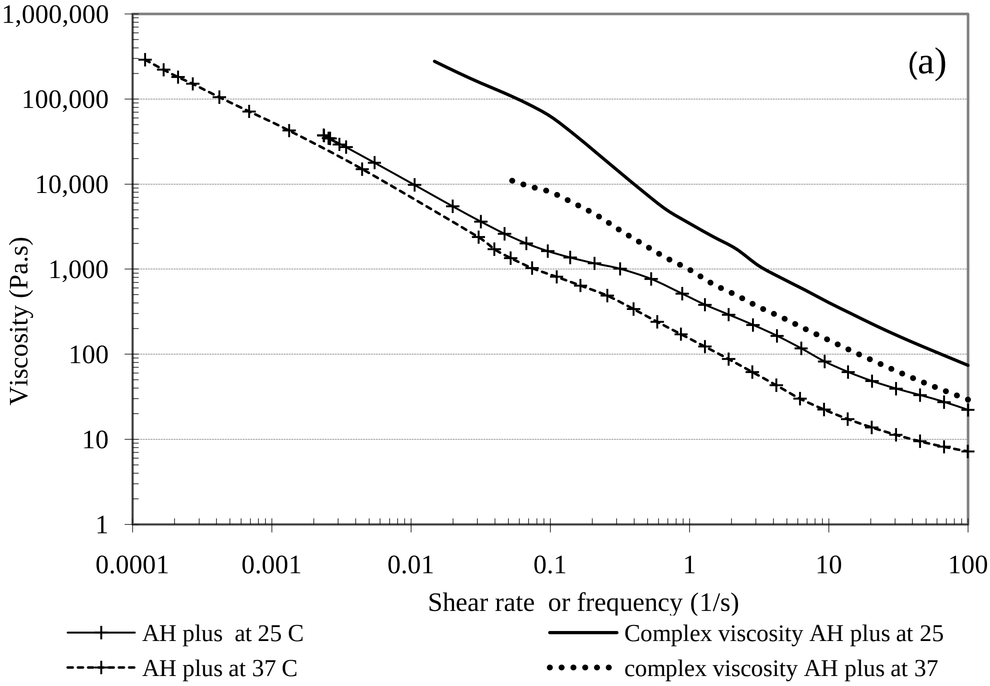


**Fig2 A**. Comparison of complex viscosity and steady shear viscosity of AH plus at two temperatures

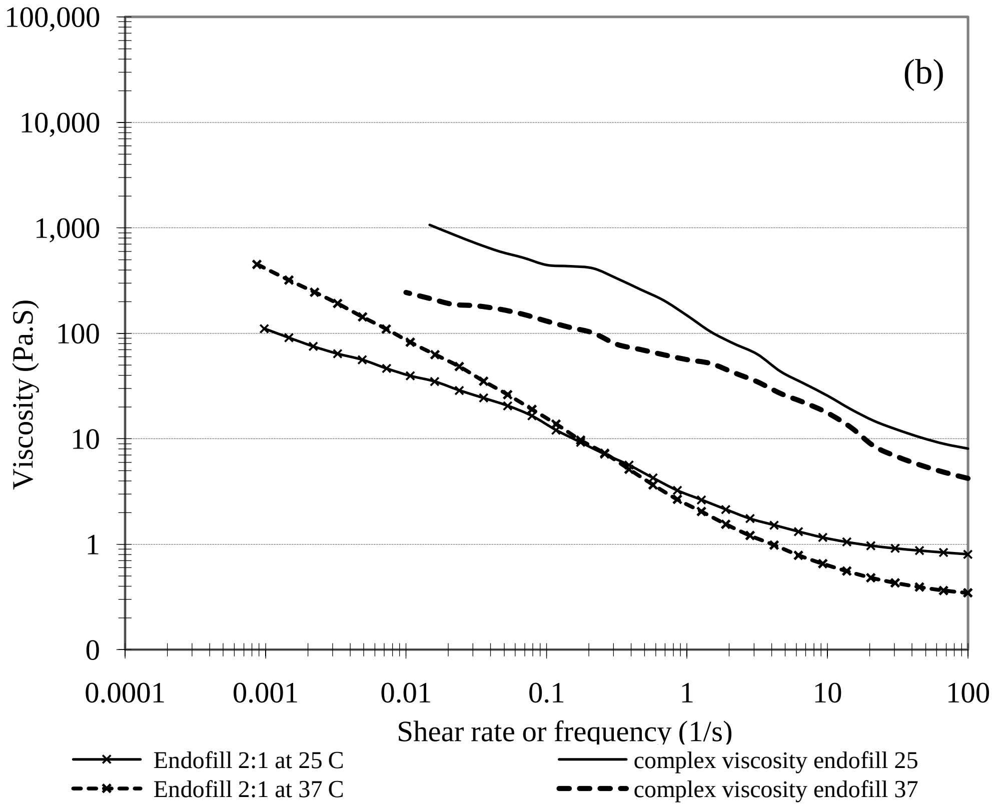


**Fig2 B.** Comparison of complex viscosity and steady shear viscosity of Endofill at two temperatures

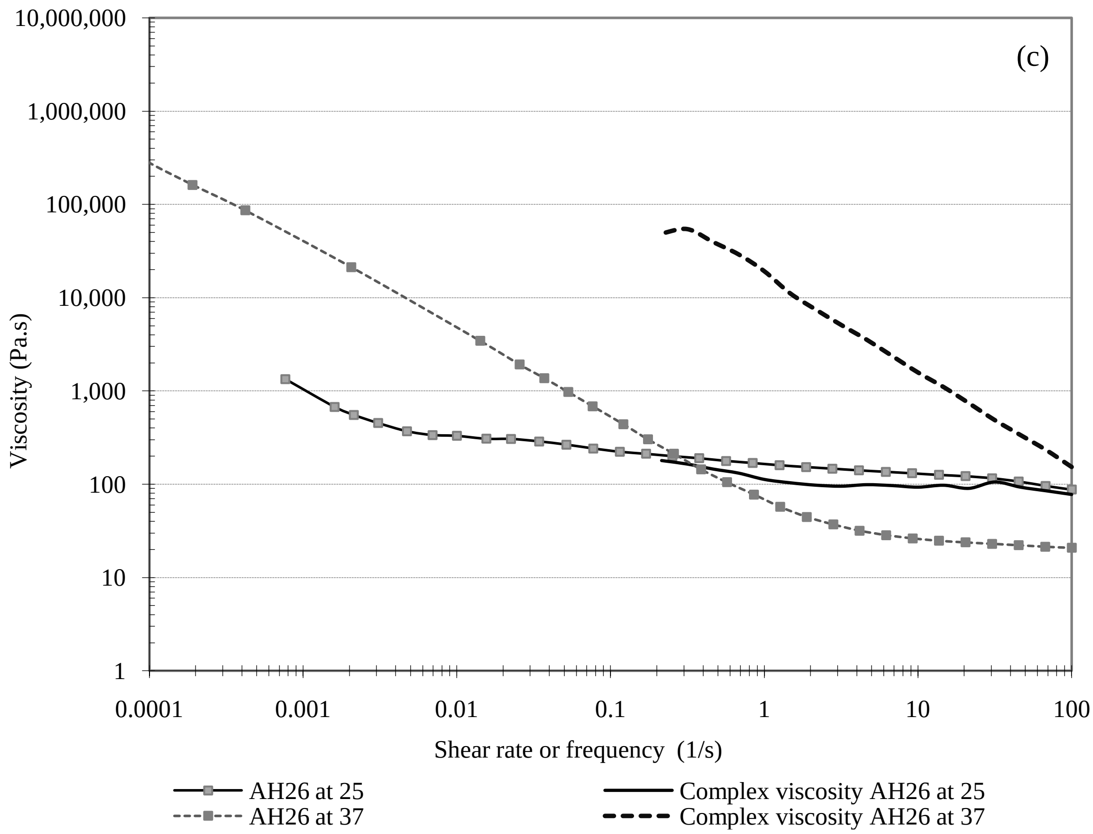


**Fig2C.** Comparison of complex viscosity and steady shear viscosity of AH 26 at two temperatures

## DISCUSSION

This study was the first investigation about the viscoelastic behavior of endodontic sealers by using a high performance strain-controlled rheometer which could calculate storage shear modulus, loss shear modulus, and dynamic viscosity. In the previous study, the flow rate or viscosity has been reported as an indicator of the rheological properties of endodontic sealers [1,6,7,9]. Therefore, the results of the present study could not be compared with other studies. The sealers are usually transferred to the root canal after mixing at room temperature and therefore undergo temperature change from the room to the mouth.

Therefore, in the present study the following temperatures were selected; 25°C a representative of room temperature and 37˚C as mouth temperature.

The MCR 300, Anton-Paar rheometer was used in this study.The instrument included a software that could calculate and plot the storage shear modulus, loss shear modulus, and dynamic viscosity from the measured torque and phase angle. In order to investigate the viscoelastic changes of the endodontic sealers as a function of the frequency, a strain sweep test was first performed in a preliminary experiment. Most of the experimental sealers exhibited a linear shear module under the shear strain of 0.5%, in which the shear module was independent of the applied strain amplitude. Therefore, the frequency sweep measurements were carried out at 0.1% of deformation for all sealers.The storage modulus of AH Plus was higher than its loss modulus over the entire experimental frequency range at two temperatures (Fig 1a).

This result indicated a dominant elastic solid-like behavior of this sealer over the examined frequency that means this sealer behaved like viscoelastic solids. This finding may be due to more interactions among the AH Plus particles that resulted to form a physical network in the structure of this sealer compared to the other sealers. Endofill exhibited a crossover region at the examined frequency range in which the storage modulus crossed the loss modulus in both temperatures (Fig 1b). Polymeric materials near the liquid-solid transition stage (LST) reveal this gel-like viscoelastic behavior. The nature of the LST in the polymeric materials physical or chemical cross linking of polymer chains [13]. The gel-like structure of Endofill is probably due to the very different microstructures of the chemical components of this sealer compared to the other sealers.

As shown in Fig 1c, AH26 at 25ºC behaved like liquids with a loss modulus higher than storage module (G"> G′).While at 37ºC, the sealer began to behave like an elastic solid material with G′>G″.At 37ºC AH Plus and AH26 at high frequencies exhibited a plateau in which both of their shear modules were nearly constant despite the increasing frequency. Physically, this plateau of storage modulus means that all the sealer particles participate in a physical network that result in a rubber like response in that scale of frequency. AH Plus and Endofill behaved similarly to temperature changes; as the temperature raised from 25ºC to 37ºC both of their shear modules decreased.

This reduction is most likely due to the decrease in the module of the resin part of these sealers by increase in temperature. This means that there is no considerable change in the viscoelastic behavior of AH Plus and Endofill in their transfer to the root canal after mixing at room temperature. In contrast AH26 showed an extensive change in the viscoelastic behavior when temperature raised from 25ºC to 37ºC. This behavior indicates that AH26 is much more sensitive to temperature changes.

Another rheological parameter defined in dynamic oscillatory shear tests is dynamic shear viscosity ($\eta^*$) or complex viscosity. Fig 2A-C shows the comparison of complex viscosity ($\eta^*$) and steady shear viscosity ($\eta$) of the test sealers. The steady shear viscosity was obtained in the preliminary experiment of this study [10]. According to this fig, the values of the complex viscosity $|\eta^*|$ of three sealers were greater than the steady shear viscosity ($\eta$). Complex viscosity is derived from the linear viscoelastic module that reflects the response of the structure of the endodontic sealers to the shearing motion of extremely low strength that essentially brings no change to the structure and preserves it throughout the measurement. In contrast, steady shear measurement queries the structure under prolonged shearing motion and upon shearing the structure will develop a steady structure consequent to the strength of the shear. Since the steady shear viscosity of the endodontic sealers is smaller than the complex viscosity, it could be concluded that the shearing motion distorts the solid-like structure of the endodontic sealers and transforms it into a less flow resistant structure. A decrease in the shear viscosity with an increase in the temperature and shear rate (fig 2A-C) was also detected. For polymeric suspensions, we expect a decrease in viscosity with an increase in the temperature and shear rate [14]. Therefore, in this study the three test sealers exhibited a polymeric suspension-like behavior. This finding is in correspondence with our previous study [10].

This study was a preliminary investigation about the viscoelastic behavior of endodontic sealers and more investigations are needed to determine the rheological properties of different endodontic sealers in clinical situations.

## CONCLUSION

According to the results of this study, AH26 at 25ºC behaved like liquids while at 37º C was an elastic solid-like material. In both test temperatures, AH Plus behaved like viscoelastic solids and Endofill exhibited a gel-like viscoelastic behavior.

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**REFERENCES**

1- Lacey S, Pitt Ford TR, Watson TF, Sherriff M. A study of the rheological properties of endodontic sealers. Int Endod J. 2005 Aug;38(8):499-504.

2- Negm MM, Lilley JD, Combe EC. A study of viscosity and working time of resin-based root canal sealers. J Endod. 1986 Oct;11(10):442-5.

3- Craig RG, Powers JM. Restorative dental materials. 11th ed. St.Louis, MO: Mosby Inc.; 2002. p. 231-51.

4- McCabe JF, Bowman AJ. The rheological properties of dental impression materials. Br Dent J. 1981 Sep 15;151(6):179-83.

5- Lee JH, Um CM, Lee IB. Rheological properties of resin composites according to variations in monomer and filler composition. Dent Mater. 2006 Jun;22(6):515-26.

6- Bernardes RA, de Amorim Campelo A, Junior DS, Pereira LO, Duarte MA, Moraes IG et al. Evaluation of the flow rate of 3 endodontic sealers: Sealer 26, AH Plus, and MTA Obtura. Oral Surg Oral Med Oral Pathol Oral Radiol Endod. 2010 Jan;109(1):e47-9.

7- Kaplan AE, Ormaechea MF, Picca M, Canzobre MC, Ubios AM. Rheological properties and biocompatibility of endodontic sealers. Int Endod J. 2003 Aug;36(8):527-32.

8- Vermilyea SG, Huget EF, De Simon LB. Extrusion of rheometry of fluid materials. J Dent Res. 1979 Jul;58(7):1691-5.

9- Lacey S, Pitt Ford TR, Yuan XF, Sherriff M, Watson T. The effect of temperature on viscosity of root canal sealers. Int Endod J. 2006 Nov;39(11):860-6.

10- Khedmat S, Momen-Heravi F, Pishvaei M. Rheological properties of endodontic sealers: the effect of time,temperature and composition. Iran Polym J. 2012;21:445-50.

11- Lee IB, Son HH, Um CM. Rheological properties of flowable, conventional hybrid, and condensable composite resins, Dent Mater. 2003 Jun;19(4):298-307.

12- Alfredo E, de Souza ES, Marchesan MA, Paulino SM, Gariba-Silva R, Sousa-Neto MD. Effect of eugenol-based endodontic cement on the adhesion of intraradicular posts. Braz Dent J. 2006;17(2):130-3.

13- Pishvaei M, Graillat C, McKenna TF, Cassagnau P. Rheological behaviour of polystyrene latex near the maximum packing fraction of particles. Polymer. 2005;46:1235-44.

14- Macosko CW. Rheology principles measurements and applications. 1st ed. Canada: Wiley-VCH;1994. p. 470-2.