Evaluation of Rheological Properties of Two Elastomeric Impression Materials during Working Time

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Abstract: Objectives. Evaluation of rheological properties of two elastomeric impression materials, in their light and medium consistencies in terms of: viscosity, flow and development of elasticity, and their relation to the working time. Materials:Two elastomeric impression materials were used; one polyether material, and one addition silicone (VPS), both in light and medium (regular) consistencies. Methods: Oscillating rheometer (Bohlin rheometer) in cone-plate configuration was used to monitor the viscosity, phase angle, tan delta, and elastic modulus throughout the working time recommended by the manufacturer. Also, the shark fin device was used to evaluate the flow properties of the tested materials. The height of the shark fin (measured by a micrometer) indicates the flowability of the material. Results: It was found that the light-bodied polyether material maintained the lowest viscosity values as well as the most viscous (plastic) behavior through the working time, compared to the other material-consistency combinations (*$P \leq 0.05$*). The medium-bodied polyether showed the highest viscosity and the most rigid (elastic) behavior among the tested material-consistency combinations (*$P \leq 0.05$*). The shark fin device results were consistent with the rheological parameters for most groups. It reflected the combined effect of the material's viscosity as well as its plasticity.

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1. Introduction

Since the introduction of elastomeric impression materials into the dental field, they have been the materials of choice for making accurate and reliable impressions, especially addition silicones and polyethers. Addition silicones produce highly acuurate impressions because they reproduce fine surface detail, and have excellent elastic recovery and dimensional stability (Mandikos, 1998). On the other hand, polyethers are known for their intrinsic hydrophilicity and thixotropic behavior (Perry et al, 2006). All elastomers are supplied as two components: base and catalyst, which require mixing before being used, this is usually influenced by the rheological properties of the pastes (Kikuchi, 1990). Insertion into patient's mouth should be done within the working time determined by the manufacturer.

Viscosity values of impression materials are most significant during the working time stage; if the viscosity is too low, the material will either run out of the tray or will not keep intimate contact with the impression site. If the viscosity is too high at the time of placement, it might not record fine details (Reisbick, 1973).

Studies were conducted to determine the suitable working time of elastomers by measuring changes in viscosity as a function of time, after the active components are mixed. However, these studies gave no reliable indication of the development of elastic properties within the test materials (McCabe and Carrick, 1989).

It should be noted that "the working time of elastomers" as defined by ISO is "the period of time between the start of mixing and the commencement of the development of elasticity and the loss of plasticity" (Pae et al, 2008). Once elasticity is developed, seating of the impression may induce elastic strain, which upon release would result in a distorted or inaccurate impression. Some of these strains would be released immediately and others would be released during storage of the impression before pouring the cast (Reisbick, 1973).

This means that some rheological characteristics, other than viscosity and flow, are more valuable for determination of working time. That is because they give an indication of the development of elasticity within a viscoelastic material undergoing setting. These characteristics include phase angle, tan delta, and elastic modulus (McCabe and Arikawa, 1998).

The phase angle is the phase shift/lag between resultant strain and applied sinusoidal stress (Saucier and Dealy, 2000). For viscoelastic materials, it lies between 0π (for ideal elastic solids) and 90π (for ideal viscous fluids) (Ram, 1997 and Jones, 1999).

The term 'complex modulus' (G^*) is used for viscoelastic materials, which consists of elastic/*storage* modulus (G') and viscous/*loss* modulus (G'') (Li et al, 1997). The ratio between the loss modulus to the storage modulus is known as "tan δ" or loss tangent (Berg et al, 2003).

$$\tan \delta = G'' / G'$$

Both the elastic properties and the viscosity of the material affect its 'flow'. A simple device, the shark fin device (3M ESPE, Seefeld, Germany), was designed to monitor flow of the impression materials during working time (Balkenhol et al, 2007).

Thus, the current study aimed to evaluate the rheological properties of two elastomeric impression materials, throughout their working times, in terms of viscosity, phase angle, tan delta, and elastic modulus, in addition to evaluation of flow using the shark fin device.

2. Materials and methods

Materials:

The materials used in this study are listed in (Table 1). Two commercially available hydrophilic impression materials were used in the study; one polyether material, and one addition silicone (VPS), both were used in light and medium consistencies.

All consistencies of the VPS material as well as the light consistency of the polyether material were supplied in plastic cartridges (50 ml) to be used with static automixing device with the corresponding mixing tips. While, the medium consistency of polyether was supplied in flexible bags (360 ml) to be used with dynamic (mechanical) mixing machine (Pentamix, 3M ESPE, Seefeld, Germany) and its corresponding dynamic mixing tips with rotating spirals.

Methods:

1. Evaluation of the rheological properties:

Bohlin rheometer (Bohlin Instruments Ltd, Great Britain) in cone-plate configuration was used for measuring the rheological properties. Viscosity, phase angle, tan delta and elastic modulus were the chosen parameters to represent the rheological properties of the materials throughout working time.

Oscillatory mode was used to monitor the onset of elasticity (McCabe and Carrick, 1990), where a stress value of 3 Pa was applied in a sinusoidal fashion. The frequency used was 1 Hz (Balkenhol et al, 2007 and German et al, 2008). Since the study was more concerned with the rheological properties throughout the working time, the temperature was adjusted at 25^0C ± 1^0C because it is common to measure the working time at room temperature, unlike the setting time which is usually measured at mouth temperature (Anusavice, 2003).

The materials were mixed using the corresponding mixing device and dispensed on the plate of the rheometer. The cone was lowered on top of the sample and the excess material was removed from around the cone so that the material just filled the gap between the upper and the lower elements.

The cone was 4 cm in diameter and the cone angle was 4^0. The gap between the center of the cone and the lower plate was adjusted to be 150 µm, to ensure a standardized volume of the tested material.

For all materials, measurement of the rheological parameters started one minute after mixing and lasted for three minutes. The values of viscosity, phase angle, tan delta, elastic modulus were recorded at intervals of eight seconds.

2. Evaluation of flow (by the shark fin test):

The flow of both light and medium viscosities of both materials was measured using the shark fin device (3M ESPE, Germany) (Figure 1).

The shark fin device consists of upper and lower components: the lower component has a central depression surrounded by a split ring for incorporating about 7 ml of the impression material.

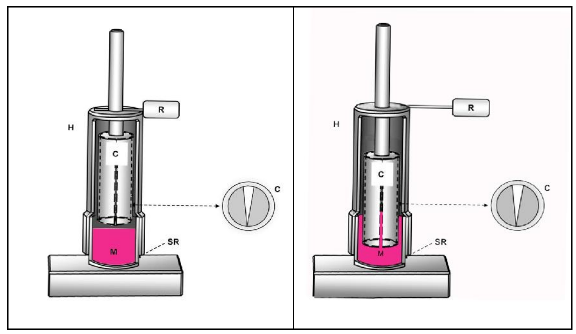

Figure (1): Schematic drawing of the shark fin device: (Left): Before removing the release pin, (Right): After removal of the release pin and flow of the material inside the slit. (SR: split ring, M: impression material, H: hollow cylinder, C: solid cylinder, and R: releasing pin to suspend the solid cylinder inside the hollow cylinder).

The upper component consists of a hollow cylinder that fits exactly around the ring of the lower part and it has a central hole at its upper end. The upper component encloses a solid cylinder with a vertical slit at its lower end, this slit extends vertically up to a distance of 32 mm. The slit was 2 mm wide at its outer end and extends horizontally in a V-shape to a distance of 1.8 mm. The upper end of the solid cylinder has a narrow rod that extends through the central hole of the hollow cylinder. The solid cylinder is suspended in its position by means of a horizontal releasing pin penetrating the narrow rod just above the central hole. When the releasing pin is removed, the solid cylinder will be free to move downwards under its own weight (147 g) which simulates the pressure applied on the wash impression material during seating of the tray

(Balkenhol et al, 2007), and sink into the material inside the split ring (Figure 1).

The test was performed at two times, one minute after the start of mixing, and then at the end of the working time recommended by the manufacturer, which was 2½ minutes for the addition silicone material and 2 minutes for the polyether material. Five samples were made for each consistency of each material at both time intervals.

The samples were measured at 60 minutes after the start of mixing, using a digital micrometer with 0.001 mm resolution (Digimatic Micrometer, Mitutoyo, Japan) (German et al, 2008). For each sample, the mean of five measurements was taken.

Table (1): The materials used in the study

Material	Consistency	Chemistry	Batch number	Manufacturer	Working time(min,s)
Impregum Garant L Duosoft	Light	Polyether	70201111385	3M ESPE, Seefeld, Germany	2 min,30s
Impregum Penta Soft	Medium		70201130054		2 min
Aquasil Ultra LV	Light	Addition silicone	080517	Dentsply, Caulk, USA	2 min
Aquasil Ultra Monophase	Medium		080131		2 min

3. Statistical Analysis:

Regression analysis using repeated measures Analysis of Variance (ANOVA) was used for studying the effect of material, consistency, time and their interactions on the rheological properties. Regression analysis using Three-way Analysis of Variance (ANOVA) was used for studying the effect of material, consistency, time and their interactions on means shark fin data. Tukey's post-hoc test was used for pair-wise comparison between the means when ANOVA test was found significant.

The significance level was set at $P \leq 0.05$. Statistical analysis was performed with SPSS 16.0 (Statistical Package for Scientific Studies) for Windows (SPSS, Chicago, IL, USA).

3. Results

Figure 2 shows the mean values of the tested rheological parameters. Figure 3 shows the variation of the rheological properties with time, where zero time corresponds to one minute after mixing where measurement was started. Figure 4 shows the mean values of shark fin heights.

There was a remarkable difference between the rheological parameters of the light and the medium consistencies of the polyether material, unlike the addition silicone, where there was no significant difference between the rheological properties of both consistencies at the end of working time.

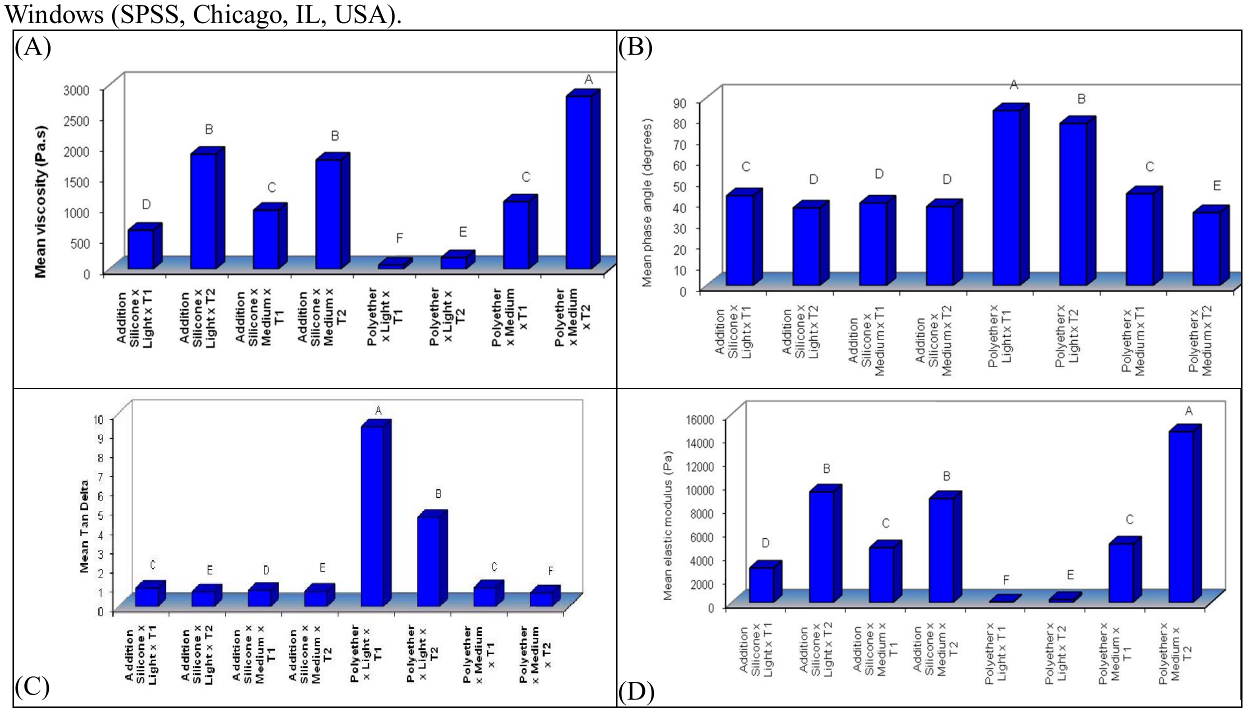

Figure (2): Mean values of (A): viscosities, (B): phase angles, (C): tan delta, and (D): elastic modulus of the different combinations of material type, material consistency, and time. (Similar letters indicate mean values not significantly different (*$P \leq 0.05$*) by ANOVA and Tukey tests).

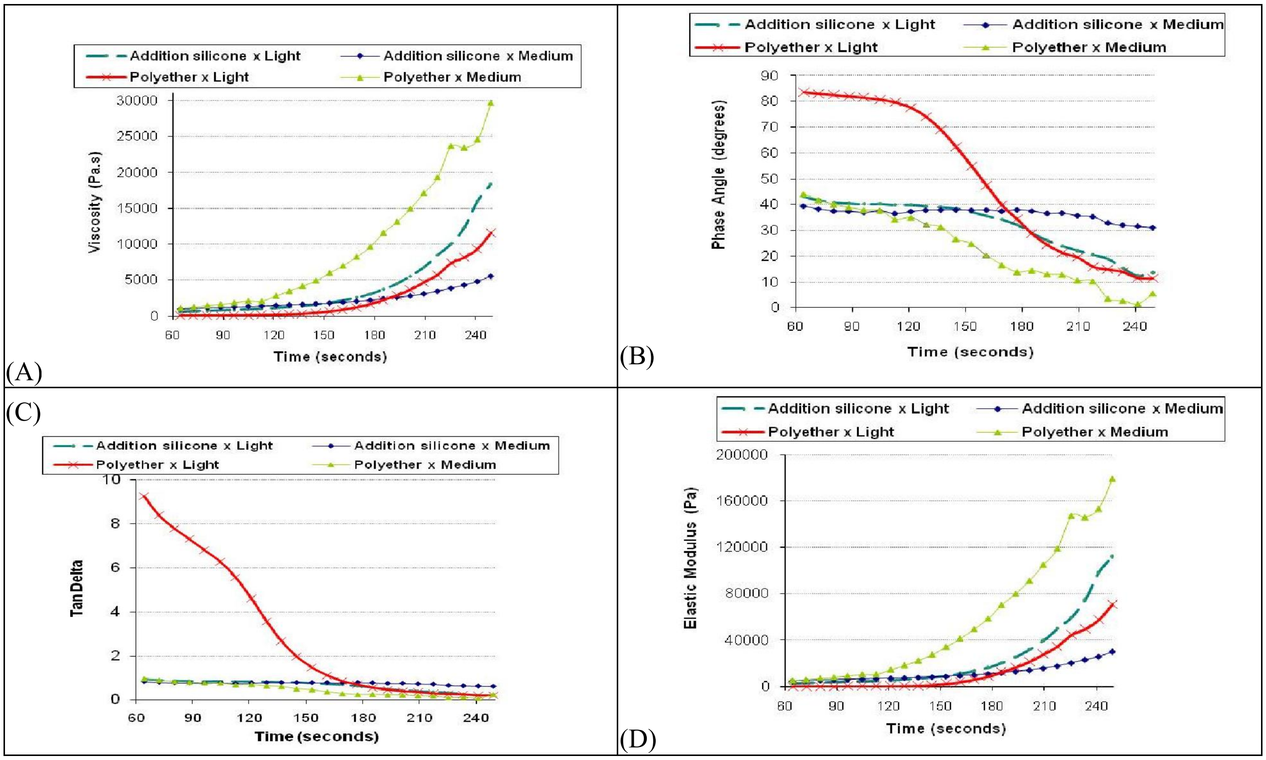

Figure (3): Change in (A): viscosities, (B): phase angles, (C): tan delta, and (D): elastic modulus of the two materials and two consistencies with time.

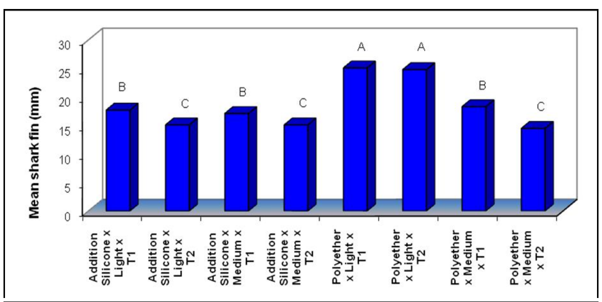

Figure (4): Mean shark fin heights of the different combinations of material type, material consistency, and time. (Similar letters indicate mean values not significantly different (*P ≤ 0.05*) by ANOVA and Tukey tests).

The results of viscosity values showed that the light bodied polyether had extremely low viscosity both at the beginning and at the end of the working time, on the contrary, the medium bodied polyether had the highest results. The light and the medium consistencies of the addition silicone were not significantly different at the end of the working time (Figure 2-A). Viscosity values increased as time elapsed, but the rate of increase was much higher after the end of the working time recommended by the manufacturer (Figure 3-A).

Phase angles were significantly different for material-consistency-time combinations, with the highest values for the light bodied polyether (Figure 2-B). This finding was the same for tan delta values (Figures 2-C). Results of phase angle and tan delta followed the same pattern, where values were high at the start of measurement, indicating fluid-like behavior, and decreased afterwards to approach zero, indicating solid-like behavior (Figure 3-B and 3-C).

Elastic modulus values showed the highest values for the medium-bodied polyether. Light bodied polyether showed extremely low values of elastic modulus (Figure 2-D).

The shark fin height of the light bodied polyether was the highest, with no significant difference between the values at the beginning and the end of the working time. However, for the other material-consistency combinations, the shark fin height decreased when the test was performed at the end of the working time (Figure 4).

4. Discussion:

1. Evaluation of rheological properties:

The rheological or flow characteristics of impression materials are major determinants in the handling properties and adaptation to the soft and hard tissues (Martinez et al, 2001).

The Bohlin controlled stress rheometer offers a convenient means of monitoring a range of rheological properties throughout the working time of elastomers (Berg et al, 2003). When materials are undergoing setting, their rheological characteristics change markedly with time. Thus, it becomes necessary to work at constant frequency and torque and monitor the varying strain (McCabe and Arikawa, 1998).

The results of the viscosity showed that the medium consistency of polyether material was significantly more viscous than both consistencies of the addition silicone at the corresponding time of measurement. On the other hand, the light consistency of polyether showed extremely low viscosity values, far below that of the addition silicone (Figure 2-A). The manufacturer of the “soft” polyethers used in this study claimed that the amount of fillers in the light consistency was reduced to decrease the final stiffness of the set material. This fact was confirmed by (Carlo et al, 2010) who reported that this light-bodied polyether had extremely low filler content (4.04%) compared to other tested materials which ranged between 23-39%.

As expected, the medium-bodied materials showed higher viscosity values than light-bodied materials due to their higher filler content. However, results revealed different behaviors for the materials tested. For the addition silicone materials, the viscosity of the light consistency was about 90% of that of the medium consistency. However, the polyethers showed a greater difference between the two consistencies where the light viscosity was about 6% of that of the medium consistency (Figure 2-A). This observation may be explained by the fact that the difference in filler loading of the light and medium consistencies of addition silicone materials is not as high as that of polyether materials, as mentioned previously.

The viscosity of elastomers during setting is affected by several factors: the molecular weight and the molecular weight distribution of the organic matter, the filler content, the base-catalyst ratio, and the rate of polymerization (Vermilyea et al, 1980). The viscosity values increased with time due to the ongoing polymerization reaction and the associated increase in molecular weight (Figure 3-A). However, the rate of increase was slow during the working time, allowing for what is called ‘induction period’, afterwards, the rate of increase in viscosity was further increased. It was noticed that the induction period of the light-bodied polyether extended to about 2½ minutes after mixing which exceeds the working time recommended by the manufacturer. On the other hand, the medium-bodied polyether showed a considerable rise in viscosity after 2 minutes from mixing, although the working time stated by the manufacturer was 2¾ minutes.

The phase angle was chosen as a parameter for determination of the development of elasticity, as it indicates the relative elastic and viscous contributions to the material’s behavior. A phase angle of 90° indicates the absence of elasticity, while a phase angle of 0° indicates that a material is behaving in a pure elastic manner (McCabe and Carrick, 1990 and McCabe and Arikawa, 1998).

The phase angle of the light-bodied polyether was found significantly higher than all other material-consistency combinations (almost double their values) (Figure 2-B). This agrees with the manufacturer’s claim that this material has exceptional flow properties, as reflected upon its ability to behave in a viscous (plastic) manner (Perry et al, 2006).

By monitoring the values of phase angle with time, there was a decrease in phase angle, indicating more elastic behavior of the materials, due to the ongoing cross-linking reaction. It was also observed that each of the tested materials (except for the medium-bodied addition silicone) had a clearly defined working time known as ‘induction period’, where phase angle values were slowly decreasing. This was followed by a rapid drop in the phase angle indicating sharp set of the materials and a rapid transition from the plastic condition to a more elastic behavior (Richter et al, 2004, and Powers and Sakagushi, 2006). This sharp set was most clear in case of light-bodied polyether.

The loss tangent (tan δ) gives an indication of the energy loss in the material during deformation, it is the ratio of the loss modulus to the storage modulus, and it is important in the evaluation of the viscoelastic properties of materials. Higher values of tan δ indicate higher energy loss and more viscous behavior, while lower tan δ values indicate more elastic behavior (McCabe and Arikawa, 1998).

The highest tan delta value was obtained by light-bodied polyether (Figure 2-C). This may also be attributed to the high viscous polymer volume fraction, on behalf of the very low filler content.

Regarding the effect of the time of measurement on tan delta values, it was found that the tan delta was higher at the beginning of the working time than at the end. This is probably because of the ongoing cross-linking reaction which decreased the viscous behavior and increased the elastic one. The rate of decrease in tan delta also suggests the presence of induction period and snap set behavior (Figure 3-C). This was more obvious for polyethers especially the light consistency, which comes in accordance with the results of (Mcabe and Arikawa, 1998) who reported that for Impregum (polyether material), the decrease of tan δ with time is initially relatively small compared with that of the other materials. Polyether materials had relatively long induction periods during which the tan δ remained almost unchanged. Not only did the light-bodied polyether material give an induction period after being mixed, but also its tan δ during this period is markedly greater than that of addition silicone material. It follows from these results that the polyether elastomer remains plastic for some time after being mixed.

The modified "soft" light-bodied polyether which is characterized by its high flow and low filler content showed the lowest values of elastic modulus, despite the fact that polyether is known for its high stiffness (Figure 2-D). Meanwhile, the medium-bodied polyether did not differ from the medium-bodied addition silicone at the start of mixing, but it showed significantly higher elastic modulus at the end of working time and throughout the setting phase. This observation was also supported by (Berg et al, 2003) who reported that even the softer versions of medium-bodied polyethers exhibited greater stiffness than the same consistency of addition silicones.

Most elastomers set through a combination of chain extension and cross-linking. Both of these processes cause an increase in elasticity (McCabe and Carrick, 1989). Since the setting reaction starts immediately upon mixing and progresses throughout the working and setting times, therefore, it was logic to find higher elastic modulus values at the end of working time than that at the beginning of working time (Figure 3-D).

2. Evaluation of flow (by shark fin test):

By measuring the flow of the materials up into a standard sized slotted-notch, under a constant load, the shark fin test theoretically allows the evaluation of the flow properties of the impression material, with the hope that these data can be extrapolated to the intra-oral situation (Balkenhol et al, 2007).

It was not possible in the current study to correlate the results of the shark fin test with the other rheological parameters, due to the fact that correlation test requires that both tests are performed on the same sample, which was practically impossible. However, when comparing the results of the shark fin test for the different material-consistency-time combinations with the results of the other rheological properties, both were found consistent with one another.

When comparing the shark fin heights of the different groups, it was noted that the light bodied polyether material showed the highest results, both at the beginning and at the end of the working time. This may be attributed to the high flow of the light consistency of the 'soft' polyether (Figure 4). This is consistent with the previously mentioned finding that the light-bodied polyether has more pronounced viscous (plastic) behavior compared to all other material-consistency combinations, as evidenced by its high phase angle and tan delta, and its low viscosity and elastic modulus (Figure 2).

Also when comparing between the shark fin results of the two consistencies, the light consistency showed higher fin heights than the medium consistency (Figure 4), which is also consistent with the rheological findings.

The time of performing the shark fin test had an effect on the shark fin height, where the shark fin heights obtained one minute after mixing were greater than those obtained at the end of working times (Figure 4). This also agrees with the results of viscosity and elastic modulus which increased with time, while the phase angle and tan delta decreased with time (Figure 3).

According to these results, it is evident that the shark fin test results gave a reliable relevance to the rheological properties. However, an exception to this relevance was found for the light-bodied addition silicone which was found to show no significant difference in fin height at the beginning and the end of working time (Figure 4). This is not consistent with the fact that the time of measurement had a significant effect on the rheological properties of the light-bodied polyether material. Significantly higher viscosity and elastic modulus values as well as lower phase angle and tan delta values were obtained at the end of working time (Figure 2).

Summary:

Different rheological properties of impression materials were evaluated throughout the working time recommended by the manufacturer. Oscillating rheometer (Bohlin rheometer) in cone-plate configuration was used to monitor the viscosity, phase angle, tan delta, and elastic modulus. Phase angle, tan delta, and elastic modulus were indicative of the development of elasticity throughout the working time. Also, the shark fin device was used to evaluate the flow properties of these materials. This device allows flow of the freshly mixed materials up into a narrow slit, forming a shark fin-like projection. The height of the shark fin (measured by a micrometer) indicates the flowability of the material.

When comparing the viscosity of the different materials' consistencies tested, it was found that the light-bodied polyether was the one with the lowest viscosity, both at the start and at the end of working time. It was also found that light-bodied polyether

maintained the most viscous (plastic) behavior through the working time, compared to the other material-consistency combinations, as indicated by its highest phase angle and tan delta and its lowest elastic modulus values. On the other hand, the medium-bodied polyether was more rigid than the medium-bodied addition silicone.

The shark fin device results were consistent with the rheological parameters for most groups. It reflected the combined effect of the material's viscosity as well as its plasticity. This is because a highly viscous material will not flow easily inside the narrow slit, also, a material that has developed some elasticity will undergo elastic deformation under the load of the device rather than penetration up into the slit. The light-bodied polyether showed the highest values of shark fin height among all material-consistency combinations, which may be related to its low viscosity and elastic modulus, and its high phase angle and tan delta.

Conclusions:

The tested newly introduced light-bodied soft polyether impression material, used with the automixing system, has exceptional flow properties in terms of low viscosity and elastic modulus values, and high phase angle and tan delta values.

For the tested materials, the development of elasticity through the working time was consistent with the increase in viscosity.

It is important to use the impression material within the working time recommended by the manufacturer, where the material is still in the plastic state and exhibits low viscosity, except for medium-bodied polyether which showed premature setting, earlier than the working time stated by the manufacturer.

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