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Effect of temperature on the rheological properties of dental interocclusal recording materials

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Abstract

The purpose of this study was to compare rheological properties of six dental interocclusal recording materials and to investigate the effect of temperature on the rheological properties during setting. Five polyvinylsiloxane materials and one polyether material were investigated in this study. The strorage modulus (G') and the loss factor (tanδ) were measured from 30s after mixing during setting, using the universal dynamic spectrometer. Viscoelastic properties were evaluated by means of G' and tanδ from 5 repeats at 21°C and 33°C. Individual changes during setting were also evaluated. All data were statistically analyzed using one-way ANOVA and multiple comparison *Scheffé* test at the significance level of 0.05. The mean of G was checked at t_{set} (the setting time provided from manufacturer) and t_{300} (the end of experimental time) and the mean of tanδ was checked at t_0 and t_{set}. Whereas the increase of the G' value showed generally exponential changes at 21°C, the change of the G' value at 33°C displayed sigmoidal curves during setting. The change of loss factor tanδ during setting varied. Within the limitations of this study, dental interocclusal recording materials had different viscoelastic properties and most of the materials showed different fluidity at 21°C and 33°C.

Keywords : elastomeric interocclusal recording material, storage modulus, loss factor, viscoelastic

1. Introduction

For precise diagnosis and correct dental treatment, it is necessary to record maxillomandibular relationship and accurately transfer it to the articulator. Correct interocclusal records and proper articulation give the clinician the opportunity to make only minimal adjustments to the restorations delivered from the dental laboratory. The chosen interocclusal material can critically affect the accuracy of interocclusal registration (Michalakis *et al.*, 2004a; 2004b; 2004c).

Many materials have been used for maxillomandibular registration procedures including wax, acrylic resin, zinc oxide-eugenol pastes, modeling compound and plaster. Currently, elastomeric materials such as polyether and polyvinylsiloxane have been widely used for the same purpose. When elastomeric interocclusal recording materials are clinically used, flow characteristics of the mixed materials are necessary to reproduce a surface detail. But over clinically reasonable time periods, they must have solid-like hardness to retain the shape and strength when dental casts are articulated. This is often described as viscoelastic behavior (Mezger, 2002). The viscoelasticity of dental materials is important in the selection of suitable materials for clinical applications.

Rheological experiments are useful to investigate the time-dependent viscoelasticity of the materials. They reveal not only information about the flow behavior of liquids, but also the deformation behavior of solids. A number of rheological investigations of elastomeric materials have been reported (McCabe and Carrick, 1989; 1990; McCabe and Bowman, 1981; Eyre *et al.*, 1989; Martinez *et al.*, 2001; Ohasawa and Finger, 1986). Such studies have all been carried out at room temperature (21°C to 25°C), whereas the setting reaction occurs at the intraoral temperature in practical. McCabe and Arikawa (1998) investigated the rheological properties of 5 different elastomeric impression materials at 23°C and 25°C. They reported a significantly more rapid development of elasticity at the higher temperature. Berg and Johnson (2003) investigated the viscoelastic properties of low and medium viscosity elastomeric impression materials during setting at 33°C and the medium viscosity materials at 3 additional temperatures. They showed the temperature-dependent behavior of viscoelasticity of impression materials and also suggested the importance of obtaining results as close as possible to the intraoral temperature and of exploring the temperature

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Table 1. Interocclusal recording materials investigated in this study

Material	Lot No.	Type	Manufacturer
Blu-Mousse	BM-06059/06059	Polyvinylsiloxane	Parkell, Farmingdale, NY, USA
Silagum	560552	Polyvinylsiloxane	DMG, Hamburg, Germany
Imprint Bite	62831	Polyvinylsiloxane	3M ESPE AG, Seefeld, Germany
Registrado X-tra	591798	Polyvinylsiloxane	VOCO, Cuxhaven, Germany
Exabite II	0510041	Polyvinylsiloxane	GC Dental Products, Tokyo, Japan
Ramitec	77821	Polyether	3M ESPE AG, Seefeld, Germany

dependence of the setting reaction in more detail.

The purpose of this *in vitro* study was to compare rheological properties of six elastomeric dental interocclusal recording materials and to investigate the effect of temperature on their rheological properties during setting.

2. Material and methods

Six interocclusal recording materials were investigated in this study. Five polyvinylsiloxane materials investigated were Blu-Mousse, Exabite II, Silagum, Imprint Bite and Registrado X-tra. One polyether was Ramitec (Table 1). The base and catalyst for each material were mixed according to manufacturers' instructions and the mixed material was transferred directly to the rheometer. Working and setting time of the materials provided from manufacturers were presented (Table 2). Rheological measurements were carried out using a Paar Physica controlled-stress rheometer (Model MCR 301; Paar Physica, Stuttgart, Baden-Wüttemberg, Germany) in an oscillatory mode. A 25 mm diameter stainless steel parallel plate assembly with a gap width of 0.5 mm was used in the time-oscillation mode. The bottom plate was used to maintain the specimen at the desired temperature. All tests were carried out according to the following protocol. The material was placed between two parallel plates. The upper plate was made to run clockwise and then counter-clockwise at a given angular frequency ($\omega = 1$) and amplitude (0.02%). The upper plate was raised to a distance of 80 mm and the base and catalyst were mixed by the mixing gun and placed directly onto the lower plate. And then the upper plate was lowered into the position (gap width of 0.5 mm), the excess material was removed and an insulating cover was placed over the two plates to sustain the temperature. As the upper plate was set into oscillation, the measurements were initiated. The measurements were started at 30s after the base and catalyst was mixed. During the operation, the rheometer was computer-driven and the rheological parameters, the storage modulus (G') and the loss factor (tanδ) were plotted automatically (RHEOPLUS/32 V2.65; Paar Physica, Stuttgart, Baden-Wüttemberg, Germany). From 30s after mixing, data was collected every 5s for 300s. Measurements were repeated 5 times at 21°C and 33°C. All data were statistically analyzed using one-way ANOVA and multiple comparison *Scheffé* test at the significance level of 0.05. SPSS software (Version 10.1, SPSS inc., Chicago, Ill, USA) was used for these statistical analyses.

Table 2. Working and setting time (sec) of the materials provided from manufacturers

Material	Working time	Setting time
Blu-Mousse	30	120
Silagum	30	90
Imprint Bite	20	60
Registrado X-tra	30	40
Exabite II	45	90
Ramitec	120	300

3. Results

The storage modulus (G) and the loss factor (tanδ) of each material at 21°C and 33°C are shown in Figs. 1 to 4. The plotted points at each time are the arithmetic averages of the results of the 5 repeat runs. The kinetics of the setting reaction over the time was shown in terms of G and tanδ. The increase of G and the decrease of tanδ provided development of elasticity in the specimens. Except for Exabite II and Silagum, the increase of the G' value of all materials showed exponential changes at 21°C, on the contrary, the change of the G' value at 33°C displayed sigmoidal curves during their setting time.

The computed parameters for all materials are summarized in Table 3 to 6. The G was checked at t_{set} (the setting time provided from manufacturer) and t_{300} (the end of experimental time) and tanδ was checked at t_0 and t_{set}.

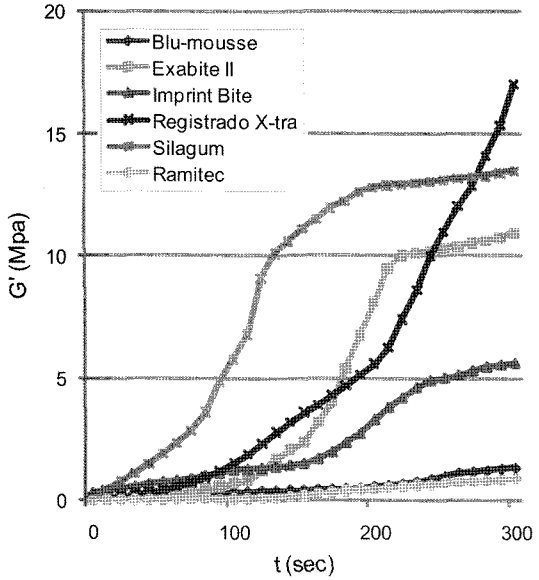

Fig. 1. The representative for storage modulus (G) at 21°C.

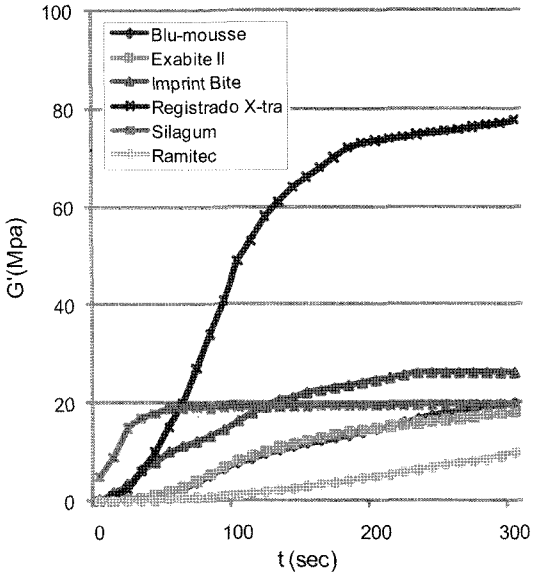

Fig. 3. The representative for storage modulus (G) at 33°C.

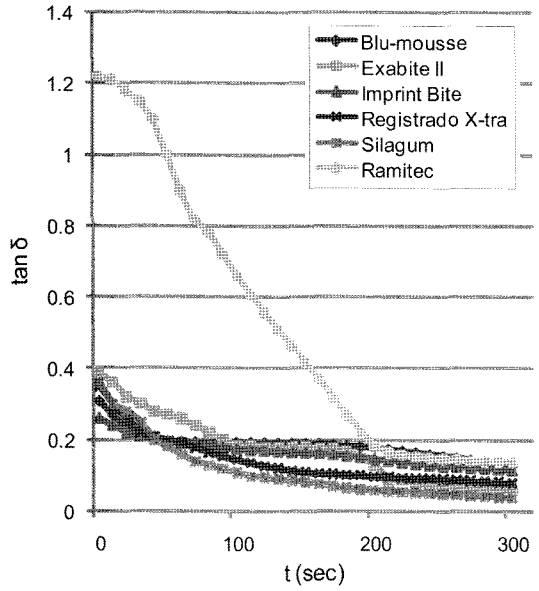

Fig. 2. The representative for loss factor (tanδ) at 21°C.

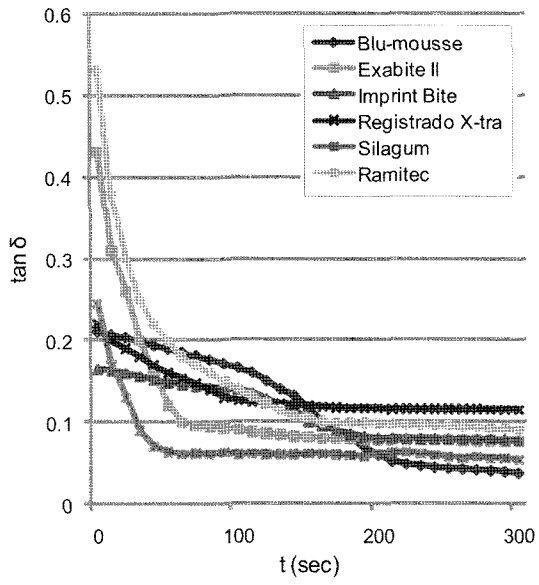

Fig. 4. The representative for loss factor (tanδ) at 33°C.

These times were measured from the initiation of the rheological measurements, which was 30 seconds after mixing the base and the catalyst as previously described. The values shown for the solid-like hardness (G') at t_{set} ranged from 0.01 (±0.01) MPa for Ramitec, to 1.94 (±0.37) MPa for Silagum at 21°C, and 2.35 (±0.56) MPa for Blu-mousse to 15.40 (±2.81) MPa for Silagum at 33°C. The storage modulus G' at t_{300} ranged from 0.72 (±0.23) MPa for Ramitec to 22.02 (±3.53) MPa for Registrado-Xtra at 21°C, and 5.42 (±1.86) MPa for Ramitec to 95.70 (±4.15) MPa for Registrado-Xtra at 33°C. The G (t_{set}) values at 21°C were compared with the values at 33°C. The G values at 33°C were significantly higher than those at 21°C ($P < 0.05$). The mean G values of the elastomeric interocclusal recording materials decreased in the following order: Registrado X-tra > Silagum > Exabite II > Imprint Bite > Blu-Mousse > Ramitec at 21°C, Registrado X-tra > Silagum > Imprint Bite > Blu-Mousse > Exabite II > Ramitec at 33°C. The one-way ANOVA test demonstrated that G values were significantly different between materials at 21°C and 33°C ($P < 0.05$).

The values shown for the tanδ at t_0 ranged from 0.34 (±0.03) for Silagum to 0.91 (±0.11) for Ramitec at 21°C, and 0.25 (±0.08) for Imprint Bite to 0.89 (±0.39) for Ramitec at 33°C. The mean of tanδ (t_0) at 21°C for polyether was significantly higher than that of polyvinylsilox-

Table 3. The storage modulus (G') at t_{set} (MPa)

		Blu-mousse	Exabite II	Imprint Bite	Registrado X-tra	Silagum	Ramitec
21°C	MEAN	0.39	0.56	0.82	0.35	1.94	0.011
	SD	0.15	0.10	0.15	0.07	0.37	0.002
33°C	MEAN	2.35	12.54	2.67	2.98	15.40	19.27
	SD	0.56	0.74	0.55	1.67	2.81	33.51

Table 4. The storage modulus (G') at t_{300}(MPa)

		Blu-mousse	Exabite II	Imprint Bite	Registrado X-tra	Silagum	Ramitec
21°C	MEAN	1.37	9.84	5.63	22.02	13.00	0.72
	SD	0.39	0.88	1.23	3.53	0.54	0.23
33°C	MEAN	15.32	14.00	16.20	95.70	17.24	5.42
	SD	3.73	0.81	4.83	4.15	2.32	1.86

Table 5. The loss factor (tanδ) at t_0

		Blu-mousse	Exabite II	Imprint Bite	Registrado X-tra	Silagum	Ramitec
21°C	MEAN	0.39	0.46	0.44	0.43	0.34	0.91
	SD	0.02	0.07	0.14	0.06	0.03	0.11
33°C	MEAN	0.27	0.45	0.25	0.44	0.41	0.89
	SD	0.06	0.17	0.08	0.25	0.31	0.39

Table 6. The loss factor (tanδ) at t_{set}

		Blu-mousse	Exabite II	Imprint Bite	Registrado X-tra	Silagum	Ramitec
21°C	MEAN	0.18	0.25	0.17	0.27	0.15	0.17
	SD	0.03	0.02	0.00	0.02	0.02	0.03
33°C	MEAN	0.14	0.09	0.16	0.18	0.07	0.08
	SD	0.01	0.01	0.01	0.02	0.01	0.05

ane materials. The loss factor tanδ at t_{set} ranged from 0.15 (±0.02) for Silagum to 0.27 (±0.02) for Registrado X-tra at 21°C, and 0.07 (±0.01) for Silagum to 0.18 (±0.02) for Registrado X-tra at 33°C.

At 33°C, most of the polyvinylsiloxane materials had reached their maximum G values by the termination of the test, whereas the polyether material showed that G values were still increasing during 300s. Multiple comparison *Scheffé* test showed that there were significant differences in G and tanδ values between polyether and polyvinylsiloxanes at 21°C ($P<0.05$). At 33°C, Registrado X-tra (PVS) showed significantly higher G values than Ramitec (PE) ($P<0.05$). Blu-Mousse, Exabite II, Silagum and Imprint Bite were comparable ($P>0.05$). Also, Registrado X-tra showed significantly higher G values than the other polyvinylsiloxane materials ($P<0.05$). Blu-Mousse, Exabite II, Silagum and Imprint Bite were comparable ($P>0.05$).

4. Discussion

Many methods have been reported for assessing rheological properties. Wilson (1966) described the use of a reciprocating rheometer which presents in graphical form the rheologial changes of the material during setting. Braden (1967) used a cone and plate viscometer. By this

method, viscosity was measured as a function of the rate of shear. These results reflect the snap set of the silicones and the more gradual setting of polysulfides. Combe and Moser (1978) used an indirect extrusion viscometer to investigate the properties of impression materials. Vermilyea *et al.* (1978) discussed the use of a capillary extrusion rheometer in relation to the properties of endodontic sealers. In this study, rheological measurements were carried out using a controlled-stress rheometer in an oscillatory mode. The controlled stress rheometer serves as a helpful instrument in the study of rheological properties during the setting of elastomers. It works on the principle of applied torque and measured deformation or strain. In addition, small changes in viscosity during setting can be detected and a quantitive value for hardness and fluidity can be obtained. This apparatus can be used at different modes including creep, oscillation and flow and various types of materials can be tested.

The storage modulus (G) and the loss factor (tanδ) of eight elastomeric interocclusal materials during setting procedure were observed at 21°C and 33°C. Jamani *et al.* (1989) suggested that the temperature of the open mouth was slightly lower than the body temperature. Thus, they suggested 33.0±0.5°C is a realistic value for assessing the properties of impression materials under oral conditions. The G was checked at t_{set} (the setting time provided from manufacturer) and t_{300} (the end of experimental time). These times were measured from the initiation of the rheological measurements, which was 30 seconds after mixing the base and the catalyst as previously described. Results for the G values of all materials at 33°C were significantly higher than the G values at 21°C, and the rate of increase of G values was rapid at 33°C. Those results were close to the earlier works (Inoue and Wilson, 1978a; 1978b; 1978c ; Omori and Arikawa, 2001).

The definition of working time by ISO (1988) is "*the period of time between the start of mixing and the commencement of the development of elasticity and the loss of plasticity.*" The determination of working and setting time has always been a problem, since they are not absolute properties and the polymerization reaction continues for a long time after the mixing has begun, in some cases it may last for hours. Ohsawa and Finger (1986) investigated working times by means of the ADA No. 19 specification test and by an oscillating rheometer method. Abuasi *et al.* (1993) developed a method for measuring both working time and setting time of elastomeric impression materials. A displacement rheometer was used to monitor the development of elasticity in setting elastomers. Jamani *et al.* (1988) used an instrument called Comprheometer to determine consistency, working time and setting time of elastomeric impression materials. The working time was measured for assessment of two-fold increase in consistency and the setting time was obtained from the development of a constant yield curve. In this study, working time and setting time of the elastomeric materials can be calculated by monitoring the development of elasticity (the storage modulus, G) and loss of fluidity (the loss factor, tanδ). In most of the materials investigated in this study, the time when G reached their maximum values was slightly longer than the setting time provided by the manufacturer.

As the setting reaction of the elastomeric materials progressed, the storage modulus (G) increased and the loss factor (tanδ) decreased. The G values (solid-like behavior) were significantly different between materials at 21°C and 33°C. At 21°C, most of the materials showed G values still increasing at t_{300}, but the G values of Exabite II and Silagum almost reached their maximum G values. At 33°C, most of the polyvinylsiloxane materials investigated in this study have reached their maximum values of G before termination of the test, whereas the G values of polyether (Ramitec) slowly increased until the end of the test. The rate at which the rheologic properties changed was markedly different for all materials. The G values of Exabite II and Silagum changed rapidly at both temperatures after the materials were mixed. This result meant that the setting of these materials occurred rapidly.

Interocclusal recording materials should have proper stiffness and the least possible plastic distortion after setting. From the results given in Figs. 1 to 4, Registrado X-tra showed significantly high G values compared with the other materials at the end of their working time. High G values at the end of the setting time indicate that the material is more resistant to deformation. On the other hand, the tanδ gives an energy loss in the material during deformation. High tanδ value indicates higher energy loss and more viscous behavior. Polyether displayed higher loss factor tanδ (t_0) and lower G values (t_{300}) than polyvinylsiloxane. Traditional polyether impression materials have been considered to be the least flexible on setting, but these results indicate that polyether may be used for mobile teeth and make less displacement to mandible in case of central relation recording as well as polyvinylsiloxane. However, there were two limitations in this study. The loss factor (tanδ) values were difficult to interpret owing to the experimental difficulty of obtaining accurate values of the moduli. These phenomena markedly occurred when the materials showed higher G values. However, this was an inevitable consequence of the low amplitude of oscillations, which had to be used to avoid going outside the linear viscoelastic region. The test was difficult to carry out faster than 30s because the upper plate was to be lowered from 80 mm into the position (gap width of 0.5 mm), the excess material was to be removed and then the insulating cover was to be placed. As working time of the interocclusal recording materials was from 20s to 45s provided from manufacturers, more studies are needed to evaluate

the properties of the materials considering the time spent for the preparation of the specimens in this study.

5. Conclusion

Within the limitations of this study, the following conclusions were drawn:

1. The interocclusal recording materials showed temperature-dependent viscoelastic properties. The G values at 33°C were significantly higher than those at 21°C.
2. Significant differences were found in G and tanδ values between polyether and polyvinylsiloxane at 21°C. At 33°C, Stonebite (PVS) and Registrado X-tra (PVS) showed significantly higher G values than Ramitec (PE).
3. Registrado X-tra exhibited significantly higher G values than the other polyvinylsiloxane materials at 21°C and 33°C. Blu-Mousse, Exabite II, Silagum and Imprint Bite were comparable.

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