Effect of preheating on the viscosity of composite resins

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Abstract

Aim: To determine the effects of time and temperature on the viscosity of preheated composite resins.

Materials and Methods: Eleven composite resins were heated to 60°C, and temperature analyses were performed at intervals of 1 min until they had cooled to 25°C. The permanent oscillatory shear test was performed at 25°C, 35°C, 50°C, and 60°C for three composite resins under a shear rate of $1s^{-1}$. One- and two-way analysis of variance were used for the analysis ($\alpha = 0.05$).

Results: There was no significant interaction between the composite resin and time ($P = 0.9304$), and only the main effect time was significantly different ($P < 0.0001$). A difference was observed between T0 and T6 ($P < 0.001$), but not after T7. The increase in temperature resulted in a viscosity reduction ($P < 0.05$). At 25°C, Beautifil II presented higher viscosity. Palfique LX5 showed a significant viscosity reduction with increasing temperature compared with the others ($P < 0.05$). For Beautifil II and Z100, there was no difference at temperatures of 50°C and 60°C, while for Palfique LX5, no statistical difference was observed at 35°C, 50°C, and 60°C.

Conclusions: Ten minutes of preheating were sufficient to reach a temperature of 60°C, reducing viscosity by at least 84%. However, 5 min after removal, the composite resin cooled to room temperature.

Clinical Significance: Preheating composite resin has potential benefits. To determine how this approach will work in clinical practice, it is important to define the effects of time and temperature in the protocol of this technique and understand its limitations.

Keywords: Composite resin; dental materials; preheating; viscosity

INTRODUCTION

Adhesive dentistry is a major component of contemporary practice that is used in virtually all clinical situations, including direct and indirect restorations. When indirect restorations are cemented, both the dental substrate and the chosen restorative material must be considered, and resin cements are currently the materials of choice for providing a bond between ceramics or resins and tooth structure.[1,2] Resin cements have low solubility in oral fluids and favorable mechanical resistance.[3] Despite the many available cementing agents, other materials have been used for this purpose, including light-polymerized composite resins.[4]

Composite resins, because of their high inorganic filler content, have greater resistance to wear compared with resin cement,[5] as well as greater stain and mechanical

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resistance.[6] However, the higher inorganic filler content of composite resins leads to a higher viscosity than that of a resin cement.[6] Heating composite resins has been used as a strategy to decrease their viscosity, reducing the cement line at the adhesive interface.[7,8] When heated to between 54°C and 70°C, the degree of conversion of the resin is similar to that of a dual-polymerizing resin cement.[4,7,8] In addition, preheating this material can reduce polymerization shrinkage[7,8] and reduce photopolymerization time by approximately 75%, resulting in a similar or better conversion than when the composite resin is light polymerized at room temperature for the manufacturer's recommendation time.[7,8] However, when the composite resin is removed from the heater at 60°C, there is significant and immediate cooling.[7,8]

Composite resins have different compositions and particle content, which leads to different degrees of fluidity after heating.[5,9,10] Although rapid heat loss can avoid pulpal damage, any delay before restoration cementation will increase viscosity, leading to a thicker cement line, preventing complete seating, and adversely affecting restoration longevity.[5,9-11] Determining the time that a composite resin takes to reach the heating temperature and then cool down to room temperature is important to define the working time and to ensure that the heated composite resin technique is optimized. Thus, the objectives of the present study were to determine the time required for different resins to reach a stable heating temperature, the cooling time required to return to room temperature, and the influence of different heating temperatures on viscosity. The research hypothesis was that time and temperature would affect the viscosity of preheated composite resins.

MATERIALS AND METHODS

Time-temperature analysis

This study analyzed the effects of time and temperature on different composite resins shade A2 [Table 1] and on their viscosity under heating with precise temperature control based on rheometer tests at the Department of Pharmaceutical Sciences (LabMac – ULEG, UnB, Brazil). The names and brands of the composite resins were hidden during testing, and all experimental tests were conducted blind and by the same researcher.

A portable device with digital temperature control was used to heat the composite resins. The temperature reported on the display was the same as the actual temperature inside the device. This was confirmed after five tests at different times and on different days with the thermal sensor (Simpla TA21, AKSO, São Leopoldo, RS, Brazil). The heating protocol was determined for this digital oven as follows: after turning on the device, the temperature was set to 60°C; this was followed by waiting for 40 min so that the device could stably reach the preset temperature. A variation of ±4°C was observed.

To determine the time required for the resins to reach this preheating temperature, each composite resin was tested individually at 60°C (±4°C). The resins were kept in a refrigerator at 5°C until the tests were performed when they were removed from the refrigerator and kept at room temperature (approximately 25°C) for 20 min until their temperature stabilized at the laboratory temperature.[12] As soon as their temperature was stable, the resins were individually weighed, and a predetermined volume (0.6 ml) was dispensed as a Ø1-cm specimen onto a glass plate and heated. The thermal sensor was positioned close to the composite resin specimens. All tests were conducted in triplicate ($n = 3$), and the means and standard deviations of the results were determined, thus obtaining the time required for each resin to reach the predetermined temperature.

As soon as the resin reached the heating temperature, the glass plate was removed, and the specimen temperature read with an infrared thermometer (Simpla TI38, 8:1, AKSO, São Leopoldo, RS, Brazil) with a detection range of −5°C–380°C. The thermometer was fixed at a distance of 8 cm from the Ø1-cm composite resin specimen on the glass plate. The temperature was analyzed at

Table 1: Characteristics and formulation of composite resins tested

Materials	Type	Manufacturer	Formulation	
			Resin phase	Filler weight % (vol %)
Beautifil II	Nanohybrid	Shofu	Bis-GMA, TEGDMA	83.3 (68.6)
Charisma Classic	Micro-hybrid	Kulzer	Bis-GMA	(61)
Charisma Diamond	Nanohybrid	Kulzer	Bis-GMA, UDMA, TEGDMA, TCD-DI-HEA	(64)
Filtek Z100	Micro-hybrid	3M ESPE	Bis-GMA, TEGDMA	84.5 (66)
Filtek Z250	Micro-hybrid	3M ESPE	Bis-GMA, UDMA, Bis-EMA, PEGDMA e TEGDMA	81.8 (67.8)
Filtek Z350XT	Nanofill	3M ESPE	Bis-GMA, UDMA, Bis-EMA, PEGDMA, TEGDMA	78.50 (63.3)
Forma	Nanohybrid	Ultradent	Bis-GMA, TEGDMA, Bis-EMA, UDMA	
Llis	Micro-hybrid	FGM	Bis-GMA, Bis-EMA, TEGDMA	77.5 (56)
Opallis	Microfill	FGM	Bis-GMA, Bis-EMA, TEGDMA	80 (72)
Palfique LX5	Supernanofill	Tukuyama	Bis-GMA, TEGDMA	82 (71)
Vittra	Nanofill	FGM	Bis-GMA, Bis-EMA, UDMA, TEGDMA	82 (60)

Bis-GMA: Bispehn ol-A glycidyl dimethacrylate, TEGDMA: Triethylene glycol dimethacrylate, UDMA: Urethane dimethacrylate, TCD-DI-HEA: Bis-(acryloyloxymethyl) tricyclodecane, Bis-EMA: Bisphenol-A ethoxylated dimethacrylate, PEGDMA: Plyethylene glycol dimethacrylate

controlled intervals of 1 min until the resin returned to room temperature (25°C) as follows: T0, immediately after removal of the heat device; T1, 1 min; T2, 2 min; T3, 3 min; T4, 4 min; T5, 5 min; T6, 6 min; T7, 7 min; T8, 8 min; T9, 9 min; and T10, 10 min after removal. The data were analyzed with the statistical software program Stata SE 15.1 (StataCorp, College Station, TX, USA). For the analysis, a two-way analysis of variance (ANOVA), with temperature as the dependent variable and resin-based composite and time as the two independent variables, as well as a *post hoc* Tukey's test, was used ($\alpha = 0.05$).

Viscosity analysis

After the first phase, 11 resins of different brands and characteristics were tested. The three heated resins with the highest fluidity were selected for viscosity testing to determine the rheological properties with the MCR 301 rheometer (Anton Paar Physica). All measurements were performed in a temperature-controlled room (25°C), and the instrument was isolated from external vibrations on a rigid support table.

A permanent oscillatory shear test was performed at 25°C, 35°C, 50°C, and 60°C for the Z100 (3M ESPE, SP, Brazil), Beautifil II (Shofu, SP, Brazil), and Palfique LX5 (Tokuyama, Via Chizzalung, Sandrigo, Italy) composite resins under $1s^{-1}$ shear rate with a rotating rheometer. The composite resins were removed from their packaging with a spatula and placed in a semicircular mold to standardize the 0.6 mL volume. The test material was dispensed on the lower plate of the rheometer, positioned with a 0.05 mm gap between the plates, and heated with the rheometer. The viscosity (Pa·s) was measured until the designated temperature was reached.

To perform the ramp analysis, the composite resins were coupled to the device at room temperature, heated to 60°C for 10 min, remained stable for 5 min more, and returned to room temperature (25°C) in 5 min, with the values defined in the time-temperature analysis. The measurements were performed at 25°C, 35°C, 50°C, and 60°C. Each composite resin was tested four times. Data were analyzed using the statistical software program Stata SE 15.1 (StataCorp). One-way ANOVA and *post hoc* Tukey's tests were used to compare the viscosity at each temperature ($\alpha = 0.05$).

RESULTS

The original data were entered into the Figshare repository (DOI: 10.6084/m9.figshare.17708510).

Eleven different composite resins were evaluated at 11 time periods (0–10 min after the removal of the heat device). As presented in Table 2, the two-way ANOVA found no statistically significant interaction between the composite

resin and time ($F = 0.77$, $P = 0.9304$), and only the main effect time was significantly different ($P < 0.0001$).

Regarding the main effect time, a difference was observed between T0, T1, T2, T3, T4, T5, and T6 (Tukey, $P < 0.001$). However, this difference was not observed after 6 min. T6 was not statistically different from T7 ($P = 0.39$); T7 was not statistically different from T8 ($P = 1.0$); T8 was not statistically different from T9 ($P = 1.0$); and T9 was not statistically different from T10 ($P = 1.0$). All experimental composite resins returned to the initial temperature (25°C) within 5 min of removal from the heating device [Figure 1].

The composite resins showed a rapid loss of temperature, losing 100% of the temperature acquired after 5 min, that is, returning to the ambient temperature (25°C). An average of -48% of the temperature was recorded after 1 min, -74% after 2 min, -87% after 3 min, and -95% after 4 min.

The results for viscosity at 25°C, 35°C, 50°C, and 60°C are shown in Table 3. For the three composite resins analyzed, the increased temperature resulted in a significantly reduced viscosity ($P < 0.05$). At the initial temperature (25°C), Beautifil II (Shofu) had a significantly higher viscosity than Palfique LX5 (Tokuyama) and Z100 (3M ESPE). Palfique LX5 (3M ESPE) started with the

Table 2: Two-way analysis of variance of dependent variable as temperature and the two independent variables (resin-based composite and time)

Source	Partial SS	DF	MS	*F*	*P*
Model	50,863.029	120	423.85857	386.65	<0.0001
Composite	10.360285	10	1.0360285	0.95	0.4925
Time	50,767.952	10	5076.7952	4631.16	<0.0001*
Composite time#	84.716705	100	0.84716705	0.77	0.9304
Total	51,128.316	362	141.23844		

*Significant difference (*P*<0.05). SS: Sum of squares, df: Degrees of freedom, MS: Mean square; *F*: F ratio
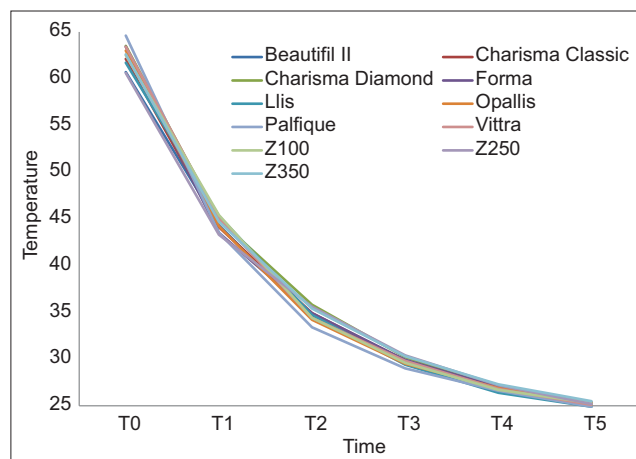

Figure 1: Cooling analysis of preheated composite resins according to time (T0–T5) in minutes after removing the heater

Table 3: Mean and standard deviation of viscosity experimental test (Pascal-second, unit of dynamic viscosity)

	Temperature (°C)			
	25	35	50	60
Beautifil II	8442.5 (1361)[a,A]	4592.5 (454)[a,B]	931 (187)[a,C]	452.5 (112)[a,C]
Palfique LX5	3012.5 (493)[b,A]	576 (58)[b,B]	272.75 (11)[b,B]	206.5 (13)[b,B]
Z100	4595 (442)[b,A]	2035 (103)[c,B]	1115.5 (118)[a,C]	690 (61)[c,C]

Different lower-case letters identify statistical difference between groups in the same columns, different upper-case letters identify statistical difference between groups in the same row

lowest viscosity, and, with increasing temperature, showed a significant viscosity reduction compared with the other two composite resins ($P < 0.05$). For both Beautifil II (Shofu) and Z100 (3M ESPE), there was no difference in the viscosity at the temperatures of 50°C and 60°C, while for Palfique LX5 (Tokuyama), no statistical difference was observed at 35°C, 50°C, and 60°C. The hypothesis that time and temperature would influence viscosity properties was accepted.

DISCUSSION

Composite resins are direct restorative materials, although they can also be used as cementing agents for indirect restorations. The indication for these materials can vary according to their composition and physical and chemical properties, and additional protocols have been suggested to improve the characteristics of the composite resins, including warming. As the use of preheated resins in dentistry has become popular, the criteria for this technique require clarification.

The composite resins can be preheated in different dry-heating devices. The temperatures for preheating composite resins described in the literature range from 54°C to 68°C, with warm-up times of 5–60 min.[5,7-13] Daronch *et al*.[12] used a device (Calset, Addent Inc.) in their study and reported that the maximum temperatures reached were 48.3°C and 54.7°C when the preset temperature of the device was set at 54°C and 60°C, respectively.[12] The digital heater used in the present study had been previously tested to avoid possible errors. After repeating the experiments on different days, the temperatures appearing on the digital display and inside the device were confirmed to be identical. Considering this, the experimental resins were subjected to heating at 60°C, with possible variations of ±4°C. A device developed specifically for this purpose facilitates this technique clinically and ensures the standardization of the preheating process, but similar heaters can also be used, provided that the temperature is controlled and remains stable until clinical use.

A wide range of heating times have been specified in the literature, varying from 5 to 60 min.[5,7-13] Eleven composite resins were tested with respect to time-temperature, and, although the materials had different compositions and characteristics, they behaved similarly in terms of heating and cooling, and a statistical difference was not found. The hypothesis tested that time and temperature would influence viscosity properties was accepted. A 10 min heating time at 60°C (±4°C) was sufficient for all the tested composite resins to reach the desired temperature, which seems a reasonable time for clinical application.

To implement this technique, the speed at which the temperature loss of each resin occurs needs to be established. According to Marcondes *et al*.,[10] composite resins preheated to 68°C showed temperature losses ranging between 45% and 61% 15 s after being removed from the heater, an 84% loss of temperature in 30 s, and a 96% loss in 60 s, suggesting that clinicians have a working time of between 10 and 15 s when seating a restoration with preheated composite resin.[10] This can also be seen in this study in part because the composite resins showed a considerable loss of temperature. However, this loss occurred over a longer time period (−48%, 1 min; −74%, 2 min; −87%, 3 min; −95%, 4 min; and −99%, 5 min), thus allowing additional working time and preserving the benefits of viscosity. Other *in vitro* and clinical studies are needed to determine an ideal working time based on these findings by considering all the criteria and sequences for the procedures. Pulpal damage has been a concern with the use of heated composite resins in dental procedures. One reason to consider the procedure safe and unlikely to damage the pulp is the rapid drop in temperature. Daronch *et al*.[13] reported that intrapulpal temperature values may not change significantly because the preheated resins are not as hot as expected when the restoration is delivered.[13] The luting procedure using preheated resins is limited to restorations of <2 mm thick because sufficient light needs to pass through to ensure complete polymerization.[4,5] Even if the technique is indicated for thin restorations, there would still be a considerable amount of excess, further reducing the chances of any pulpal damage.[5,12,13]

Regarding viscoelastic properties, restorative composite resins with different formulations can react differently to preheating.[10,14,15] The characteristics of the inorganic particles may also influence flowability, affecting the thickness of the film when used as a cement.[10] Preheating to 69°C has been reported to reduce the viscosity of restorative resin composites by 47%–92% compared with that at 37°C.[10] Therefore, thermal conductivity must also be considered in the choice of material in addition to

its handling characteristics,[15,16] including filler content, particle type, shape, size, nature of the particle surface, and spatial arrangement of the filler within the composite resin,[10] because only the relationship between viscosity and the filler content is negative.[10,17] Although there is a relationship between viscosity and temperature, no change was noted in the mechanical properties of the composite resins at the same loading of particles of different sizes.[18]

The viscosity of composite resins, however, is affected by the resin matrix formulation, the interlocking between the filler particles, and the interfacial interaction between the filler particles and resin matrix. An inverse relationship was observed between viscosity and polymerization shrinkage, depending primarily on the monomer types and ratio of the resin matrix,[16] and the filler volume has been reported to be more important than the filler weight.[15] Regarding the filler size, composite resins with a small filler size were more viscous than those with a larger filler size, even though the filler content was the same because the number of filler particles was higher for identical filler volumes. As a result, the increased surface area led to a greatly increased interaction between the resin matrix and filler particles and between filler particles.[15] The viscosity of the composite resin with a round filler seems to be much lower than that with an irregular filler because of the lower friction at similar filler sizes.[15] Diluents such as triethylene glycol-dimethacrylate and urethane dimethacrylate have lower molecular weights and lower viscosities than bisphenol A-glycidyl methacrylate.[15,16] Therefore, their low molecular weights and resultant high numbers of double bonds per unit of weight create a high degree of cross-linking and a relatively high amount of shrinkage.[16] However, manufacturers do not always disclose all the formulation details.

Resins that showed greater fluidity for handling after being heated to 60°C were selected for the analysis of viscosity. These were the nanohybrid composite Beautifil II (Shofu), micro-hybrid Filtek Z100 (3M ESPE), and supernanofill Palfique (Tokuyama). Increased temperature led to a significant viscosity reduction, ranging between 84% and 94%. However, it is expected that the preheated resin composite resins will not show viscosity values in the range of the flowable composite or resin cements.[10] For both Beautifil II (Shofu) and Z100 (3M ESPE), there was no difference in the viscosity from temperatures 50°C to 60°C, while for Palfique LX5 (Tokuyama), no statistical difference between 35°C, 50°C, and 60°C was observed. Among them, Palfique LX5 (Tokuyama) showed the lowest viscosity when heated. Although there is no difference between 50°C and 60°C in the viscosity analysis for the three resins, adopting higher temperatures will extend the working time, since the rapid loss of temperature is a limiting factor of the technique. The overall performance of the preheated composite resin technique depends on proper material selection and correct procedure because the change in viscosity upon preheating alone is not an appropriate parameter for selecting a restorative composite resin for luting purposes.[10,19] The filler content of the resins does not interfere directly with the thickness of the film,[15] and Marcondes *et al*.[10] reported that some composite resins that showed the highest reduction in viscosity were not always able to yield the thinnest films.[10]

Based on the results of this *in vitro* study, heating composite resins influences their viscosity, consistent with previous studies.[14-16,20] Ahn *et al*.[20] speculated that preheating increased the mobility of the unpolymerized monomer and allowed a better distribution of the filler particles, becoming more fluid when it was manipulated. Even though preheating alters the structure of composite resins, it seems to be beneficial because this improvement in the internal structure persists, even after cooling.[20]

Limitations of this study included that only 11 composite resins (shade A2) were investigated. Even though the preheating of composite resins affects their viscosity, future studies should be conducted on how different monomers and filler features might influence the reaction of composite resin to preheating. A working time from 30 s to 1 min may preserve their benefits and allows for better manipulation of composite resins during the luting procedure, but it seems not to be reproducible in the clinical scenario. In addition, clinical and laboratory studies should be developed to simulate the clinical procedure and determine the protocol for composite preheating.

CONCLUSIONS

The preheating of composite resins affects their viscosity; however, the following aspects are relevant to this technique. The time of 10 min at ≥60°C seems to be adequate to heat composite resins, reducing their viscosity by at least 84%. All tested resins lost 100% of the temperature gained during heating after 5 min.

Data availability
Datasets related to this article can be found at DOI: 10.6084/m9.figshare.17708510, hosted at Figshare repository.

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Conflicts of interest
There are no conflicts of interest.

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