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Effect of temperature on composite polymerization stress and degree of conversion

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ABSTRACT

Objective. To test the following hypotheses: (1) degree of conversion (DC) and polymerization stress (PS) increase with composite temperature (2) reduced light-exposure applied to pre-heated composites produces similar conversion as room temperature with decreased PS.

Methods. Composite specimens (diameter: 5 mm, height: 2 mm) were tested isothermally at 22 °C (control), 40 °C, and 60 °C using light-exposures of 5 or 20 s (control). DC was accessed 5 min after light initiation by FTIR at the specimen bottom surface. Maximum and final PS were determined, also isothermally, for 5 min on a universal testing machine. Non-isothermal stress was also measured with composite maintained at 22 °C or 60 °C, and irradiated for 20 s at 30 °C. Data were analyzed using two-way ANOVA/Tukey and Student's t-test ($\alpha = 5\%$).

Results. Both DC and isothermal maximum stress increased with temperature ($p < 0.001$) and exposure duration ($p < 0.001$). Isothermal maximum/final stress (MPa) were $3.4 \pm 2.0b/3.4 \pm 2.0A$ (22 °C), $3.7 \pm 1.5b/3.6 \pm 1.4A$ (40 °C) and $5.1 \pm 2.0a/4.0 \pm 1.6A$ (60 °C). Conversion values (%) were $39.2 \pm 7.1c$ (22 °C), $50.0 \pm 5.4b$ (40 °C) and $58.5 \pm 5.7a$ (60 °C). The reduction of light exposure duration (from 20 s to 5 s) with pre-heated composite yielded the same or significantly higher conversion (%) than control (22 °C, 20 s/control: $45.4 \pm 1.8b$, 40 °C, 5 s s: $45.1 \pm 0.5b$, 60 °C, 5 s s: $53.7 \pm 2.7a$, $p < 0.01$). Non-Isothermal conditions showed significantly higher stress for 60 °C than 22 °C (in MPa, maximum: 4.7 ± 0.5 and 3.7 ± 0.4 , final: 4.6 ± 0.6 and 3.6 ± 0.4 , respectively).

Clinical significance: Increasing composite temperature allows for reduced exposure duration and lower polymerization stress (both maximum and final) while maintaining or increasing degree of conversion.

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1. Introduction

Temperature plays an important role in the polymerization process of multifunctional monomers used in photo-activated, dimethacrylate-based dental restorative materials [1–5]. Increased polymerization temperature enhances radical and monomer mobility, resulting in higher conversion because of lowered system viscosity [3,6–9]. From a clinical standpoint, pre-heating was shown to greatly increase composite flow [10–13]. No pulpal damage is expected with pre-heating, as the difference in *in vitro* intrapulpal temperature was less than 1 °C when using composite pre-heated to 60 °C compared to composite tested at room temperature [14]. However, little information is available on the effect of preheated composites on polymerization stress.

Previous *in vitro* studies on isothermal polymerization indicated a significant increase in degree of conversion, as well as an increase in polymerization rate and conversion at maximum cure rate, when composite curing occurred above room temperature [4,5]. Moreover, pre-heated composite reached high conversion values using relatively low radiant exposures, allowing a reduction in exposure duration up 75% compared to composites polymerized at room temperature [4]. However, it has been shown that increased polymerization rate and conversion increase polymerization stress development [15,16]. Therefore, in theory, the use of pre-heated composites may result in increased stress, unless the reduction in viscosity due to heating would allow for increased viscous flow and chain relaxation, offsetting the effects of the higher conversion and reaction rate. Also, secondary transitions involving comparatively small changes in modulus as a result of temperature increase, such as side-group motions [17], could also contribute to stress relaxation.

Though information on isothermal polymerization stress is important, it does not necessarily represent what occurs clinically. With clinically available pre-heating devices, the temperature drop once composite is removed from the heating device is likely to result in lower conversion compared to that found when composite is cured isothermally at elevated temperatures [5,18]. *In vivo* tooth temperature during a restorative procedure is approximately 30 °C and a composite material originally pre-heated to 60 °C placed into a tooth preparation attained only 38 °C at the time of photo-activation [19]. Therefore, it is also important to evaluate the effect of pre-heating dental composites on polymerization stress development in a clinically realistic scenario, where composite temperature is elevated in the heating device and then lowers when in contact with the prepared tooth.

The purpose of the present study was to evaluate the influence of composite temperature and light-exposure duration on polymerization stress and degree of conversion of a commercial composite photocured either isothermally or non-isothermally, according to a clinically representative temperature scheme. Since conversion is influenced by kinetic parameters such as temperature and also radiant exposure, and polymerization stress is dependent on conversion, polymerization stress is expected to be affected by both temperature and exposure duration. The working hypotheses were (1) for a given exposure duration, increase in composite

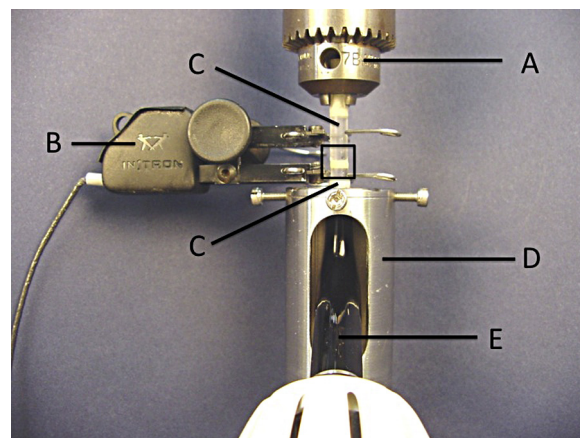


Fig. 1 – Polymerization stress test configuration. A: clamp connected to the load cell; B: Extensometer; C: Glass rods; D: Metallic fixture for light-curing end tip (E). Box: resin composite specimen. (Note: all items above were contained within a temperature-controlled chamber).

temperature will increase polymerization stress and degree of conversion and (2) for a given composite temperature, reduction in exposure duration will decrease polymerization stress and degree of conversion values.

2. Materials and methods

2.1. Polymerization stress testing

One of the ends of two 5-mm diameter glass rods, one 13-mm and the other 28-mm in length, were ground flat with SiC paper and sandblasted with alumina (250 μm). The roughened surfaces were silanated (Dentsply Ind. e Com. Ltda., Rio de Janeiro, Brazil), coated with two layers of bonding agent (Scotchbond Multi-Purpose Plus, 3M ESPE, St. Paul, MN, USA) light-cured for 30 s. The glass rods were attached to opposite fixtures of a universal testing machine (model 5565, Instron, Canton, MA, USA) by their non-treated surfaces. The long rod was attached to the actuator, while the short rod was attached to a stainless steel fixture connected to the lower clamp of the machine. This fixture had a slot allowing contact of the light tip with the glass rod (Fig. 1). A nanohybrid composite (Esthet X, shade A2, lot#0302054, Dentsply/Caulk, Milford, DE, USA) was placed between the treated glass surfaces (height = 2 mm, C-factor = 1.25, volume = 39.3 mm³).

Relevant portions of the testing assembly were maintained inside a temperature-controlled chamber (model 3119-405, Instron). During isothermal testing, composite compules (unidose) were also kept inside the chamber prior to testing in order to stabilize the material's temperature. The chamber temperature was kept at 22 ± 2 °C (room temperature, control), 40 ± 2 °C, or 60 ± 2 °C. The latter value is similar to that used in a commercial heating device (Calset™, AdDent Inc. Danbury, CT, USA). For the non-isothermal conditions, the temperature-controlled chamber was set to 30 °C, while composite compules were kept either at room temperature

($22 \pm 2^\circ\text{C}$) or in a heating device (Calset, AdDent Inc., Danbury, CT, USA), pre-set to 60°C .

Photoactivation was performed through the lower glass rod using a conventional quartz-tungsten-halogen light-curing unit (Optilux 501, Demetron/Kerr Co., Orange, CA, USA) for 20 s (control, manufacturer recommended exposure duration) or 5 s. The latter exposure duration was found to result in greater conversion when composite was pre-heated to 40 – 60°C compared to 20 s exposure at room temperature [4]. Spectral irradiance of the curing light was determined between 350–600 nm using a laboratory-grade spectral radiometer (DAS 2100, Labsphere, N. Sutton, NH, USA) having a 3-inch integrating sphere and calibrated using a NIST-traceable source. To compensate for light attenuation caused by the glass, irradiance output was increased to approximately 700 mW/cm^2 , using a “turbo” tip instead of the standard 8 mm diameter tip. The irradiance delivered from the glass rod end was 630 mW/cm^2 . The polymerization stress test was performed inside the temperature-controlled chamber, at one of the pre-set temperatures, with polymerization force being monitored for 5 min from the start of photoactivation. The distance between the glass rods was kept constant throughout the test using a feedback loop controlled by an extensometer (model 2630-101, Instron). Maximum and final (i.e., the value registered at the end of the monitoring period) force data were converted to nominal stress (MPa) by dividing force values by the cross-section area of the glass rod. Stress relaxation was determined by calculating the percentage of the corresponding maximum stress value that was observed at the 5-min interval. Five specimens were tested in each of the experimental conditions.

2.2. Degree of conversion

Degree of conversion was accessed by Fourier-transformed infrared spectroscopy. In order to monitor the conversion at the bottom surface of a 2-mm height specimen, brass rings (6 mm diameter, 2 mm high) were filled with uncured composite and placed on a temperature-controlled stage of a diamond-attenuated total reflectance (TCS-ATR) unit (Heatable Golden Gate ATR, MkII, SPECAC Inc., Smyrna, GA, USA). The ring was covered with a sheet of clear plastic matrix (0.08 mm, type D Mylar, Du Pont, Wilmington, DE, USA) and pressed to force the paste to conform to the ring dimensions and to ensure a close contact between the composite and the surface of the diamond.

Infrared spectra were collected between 1680 and 1550 cm^{-1} at a rate of one-per-second at 2 cm^{-1} resolution (FTS-40, Digilab/BioRad, Cambridge, MA, USA). The first four spectra corresponded to the uncured resin. Then, composite was light-activated using the same parameters described for the polymerization stress test and spectra continued to be collected for 300 additional seconds. Three specimens were tested per group. Monomer conversion was calculated by standard methods that utilize changes in the ratios of aliphatic-to-aromatic C=C absorption peaks in the uncured and cured states [20].

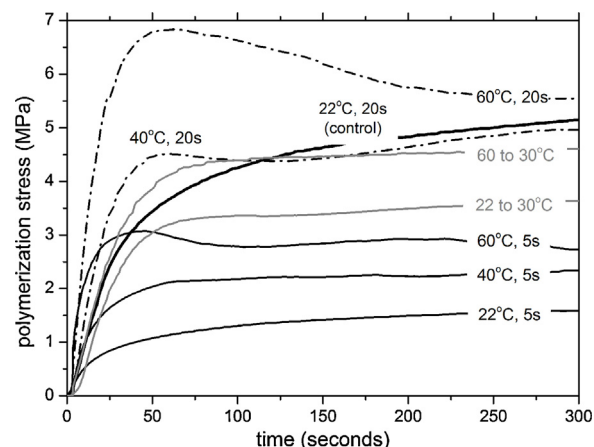


Fig. 2 – Mean values of real-time polymerization stress profiles using various composite temperatures and exposure durations.

2.3. Statistical analysis

For both isothermal and non-isothermal conditions, two-way analysis of variance (ANOVA) (composite temperature and exposure duration as main factors) followed by Tukey test were used to identify differences among the experimental groups for degree of conversion, maximum and final stress. Also, Student's t-tests compared maximum and final stress values for a given temperature and light exposure duration ($\alpha = 5\%$).

3. Results

3.1. Polymerization stress

Under isothermal conditions, both composite temperature and light exposure duration had a significant influence on maximum polymerization stress ($p < 0.001$ for both), but their interaction did not ($p = 0.204$) (Table 1). The highest maximum stress value was obtained at 60°C with 20 s exposure. Using the 20 s exposure, maximum stress at 40°C and 22°C were not statistically different. With the 5 s exposure, maximum stress also increased with temperature, but the only significant difference was with the value at 60°C , which exceeded that at 22°C .

For final polymerization stress data, only exposure duration had a significant effect ($p < 0.01$). At a given temperature, the 5 s exposure resulted in lower final stress than the 20 s exposure.

Significant differences between final and maximum stress values were observed for both exposure durations using composite at 60°C ($p < 0.01$), corresponding to stress relaxation values of 16–25% (Table 1). When composite was cured at 22°C and 40°C , no significant differences between final and maximum stress were observed, and stress relaxation was minimal ($\leq 2\%$). Stress relaxation can be observed in the stress versus time curves obtained under isothermal conditions (Fig. 2).

Under non-isothermal conditions (Table 2, Fig. 2), both maximum and final stress values were significantly higher when composite was initially pre-heated at 60°C than

Table 1 – Effects of isothermal temperature and exposure duration on polymerization stress values (mean values, with standard deviation in parenthesis).

Composite temperature (°C)	Exposure duration (s)	Maximum polymerization stress (MPa)	Final polymerization stress (MPa)	Relaxation (%) ^a
22 ^b	5	1.6 (0.2) D	1.6 (0.3)b	0
	20 ^b	5.2 (1.1) B	5.1 (1.1)a	2
40	5	2.3 (0.4) C, D	2.3 (0.4)b	0
	20	5.1 (0.2) B	5.0 (0.1)a	2
60	5	3.2 (0.7) C ^c	2.7 (0.8)b	16
	20	6.9 (0.6) A ^c	5.2 (1.0)a	25

n = 5 specimens per experimental group. Within a column, values having similar letters are not significantly different ($p < 0.05$).

^a Calculated as $1 - (\text{final stress}/\text{maximum stress}) \times 100\%$.

^b Control, room temperature and manufacturer-recommended exposure duration.

^c Significant difference between maximum and final stress for a specific composite temperature and exposure duration ($p < 0.01$).

Table 2 – Means (SD) of polymerization stress values (MPa) determined under non-isothermal conditions (pre-heated at 22 or 60 °C and cured at 30 °C for 20 s).

Initial composite temperature (°C)	Maximum polymerization stress (MPa)	Final polymerization stress (MPa)	Relaxation (%) ^a
22	3.7 (0.4) A,a	3.6 (0.4)A,a	3
60	4.7 (0.5) B,b	4.6 (0.6)B,b	2

n = 5 specimens per experimental group. Within a column (upper case) or row (lower case), values denoted by similar letters are not statistically different ($p > 0.05$).

^a Calculated as $1 - (\text{final stress}/\text{maximum stress}) \times 100\%$.

composite maintained at room temperature ($p < 0.05$ for both). For either initial composite temperature, maximum and final polymerization stress values were not significantly different ($p > 0.05$), and a minor relaxation was observed in both groups ($\leq 3\%$).

3.2. Degree of conversion

Both composite temperature and light exposure duration had a significant influence on degree of conversion ($p < 0.05$ for both), but their interaction did not ($p = 0.425$). Mean conversion values for temperature factor (%) were 39.2 ± 7.1^a (22 °C), 50.0 ± 5.4^b (40 °C) and 58.5 ± 5.7^c (60 °C) and for exposure duration were 54.6 ± 7.9 (20 s) and 43.9 ± 9.2 (5 s). Table 3 shows the mean degree of conversion values for each temperature and exposure duration tested. Statistical comparisons showed that

Table 3 – Mean (SD) degree of conversion values (%) as a function of composite temperature and exposure duration.

Temperature (°C)	Exposure (s)	
	5	20 [*]
22 [*]	33.0 (2.9) D	45.4 (1.8) C
40	45.1 (0.5) C	54.9 (0.4) B
60	53.7 (2.7) B	63.4 (1.9) A

n = 3 specimens per experimental group. Values having similar letters are not significantly different ($p < 0.05$).

^{*} Control, room temperature and manufacturer-recommended exposure duration.

reduction of light exposure duration to 5 s with pre-heated composite exhibited equal or greater conversion (%) than the control ($p < 0.001$): 45.4 ± 1.8 (22 °C, 20 s), 45.1 ± 0.5 (40 °C, 5 s), 53.7 ± 2.7 (60 °C, 5 s).

4. Discussion

The first hypothesis was validated: increasing composite temperature prior to light activation produced significantly higher polymerization stress and degree of conversion. Relative to control (22 °C/20 s), both maximum and final polymerization stress increased with temperature rise under isothermal test conditions. This finding agrees with preliminary work [9,21]. When composite temperature is raised prior to polymerization, system viscosity decreases resulting in additional monomer conversion [7,13,22–24], and, as a consequence, in higher polymerization stress.

The second hypothesis was also confirmed: reduction of exposure duration from 20 s to 5 s at a given composite temperature produced significantly lower stress and degree of conversion. Reduced exposure time results in fewer radicals being generated. However, their segmental mobility may be enhanced by the temperature rise, making more residual unsaturation sites accessible for reaction, and resulting in conversion similar to or greater than what is obtained with conventional, longer exposures given when composite is at lower temperatures [7,22].

Elevated temperatures can accelerate the photopolymerization reaction. Temperature increase improves molecular mobility, allowing for further conversion before the onset of

autodeceleration, and postponing the change in termination mechanism from one diffusion-controlled to one controlled by reaction diffusion [22,23,25]. As a consequence of the increased temperature, vitrification is postponed. The onset of vitrification occurs when the glass transition temperature approaches the curing temperature [26]. Therefore, at higher curing temperatures, final monomer conversion increases because the material can polymerize further before its glass transition temperature reaches the temperature of the polymerization reaction [3].

Prior to vitrification, polymerization stress can be relieved by viscous flow or chain relaxation [16]. In the present study, a significant stress relaxation was noticed when composite was polymerized isothermally at 60 °C using both exposure durations, and more noticeable when using the 20 s exposure. Stress reduction is believed to occur past the vitrification point, since it was found that, for this material, autodeceleration occurred before 20 s into the reaction [5]. Another report also found significant stress relaxation using pre-heated composite [21]. These findings might be associated to secondary transitions arising from side group motion when composite is maintained at relatively high temperature [17].

The results obtained under isothermal conditions, however, do not account for the temperature variation occurring either from room temperature or the pre-set temperature in the heating device to mouth temperature. To include this variable, polymerization stress under non-isothermal conditions was also tested. Surprisingly, the results indicated that polymerization stress development was not as severe as what was observed using isothermal conditions. For the composite initially at room temperature, a polymerization stress value around 5 MPa was expected, close to those found under 22 °C and 40 °C isothermal conditions. For the composite pre-heated to 60 °C and light-cured at 30 °C, stress was closer to those registered at 22 °C and 40 °C isothermal conditions. Composite temperature drops rapidly from the moment it is removed from the pre-heating device until it reaches thermal equilibrium with mouth temperature [19]. As a consequence, by the time of photo-activation the composite is not as warm as the temperature of the heating device, and the final conversion and polymerization stress values also decreased in comparison to those verified with the 60 °C isothermal specimens. In an *in vivo* study, composite temperature immediately after placement in a prepared tooth was approximately 7 °C higher when composite was pre-heated at 60 °C compared to the composite maintained at room temperature, corresponding to an approximate 4% increase in conversion [19]. This small, but significant increase in conversion might explain the 13% increase in polymerization stress observed in this study.

The present findings show a potential reduction in stress when increasing composite temperature and shortening light exposure duration, in comparison to a 20 s exposure at room temperature. Equal or higher conversion can be obtained with higher temperatures using lower energy doses than when using composite at room temperature for standard exposure duration. In terms of mechanical properties, pre-heating resin composite enhances its surface hardness [13] and showed no effect on flexural strength and polymer cross-linking estimates [27]. Moreover, no polymerizable components were lost

upon pre-heating, nor was there any degradation of monomer during different heating treatments [18].

One single brand of composite was tested in this study, and direct extrapolation of relationships observed in the present investigation to all other composites may not be valid because of differences in filler composition and chemistries. However, it is expected that the general trends observed are applicable to other dental resin formulations.

5. Conclusion

In conclusion, degree of conversion and polymerization stress significantly increases with elevated composite pre-cure temperature and decreases with lower exposure duration. Only when composite temperature was raised to 60 °C was a significant stress relaxation noted. Using a 5 s exposure (only ¼ of the manufacturer's recommended duration) on composite pre-heated to 40 °C or 60 °C resulted in a 47–55% reduction in final stress compared to using a full 20 s exposure with the composite maintained at room temperature. This finding is of major importance, as degree of conversion obtained isothermally 40 °C or 60 °C with a 5 s exposure was similar or even higher than 20 s at room temperature.

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