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Composite pre-heating: Effects on marginal adaptation, degree of conversion and mechanical properties

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ABSTRACT

Objectives. This study evaluated the effect of composite pre-polymerization temperature and energy density on the marginal adaptation (MA), degree of conversion (DC), flexural strength (FS), and polymer cross-linking (PCL) of a resin composite (Filtek Z350, 3M/ESPE).

Methods. For MA, class V cavities (4 mm × 2 mm × 2 mm) were prepared in 40 bovine incisors. The adhesive system Adper Single Bond 2 (3M/ESPE) was applied. Before being placed in the cavities, the resin composite was either kept at room-temperature (25 °C) or previously pre-heated to 68 °C in the Calset™ device (AdDent Inc., Danbury, CT, USA). The composite was then light polymerized for 20 or 40 s at 600 mW/cm² (12 or 24 J/cm², respectively). The percentage of gaps was analyzed by scanning electron microscopy, after sectioning the restorations and preparing epoxy resin replicas. DC (n = 3) was obtained by FT-Raman spectroscopy on irradiated and non-irradiated composite surfaces. FS (n = 10) was measured by the three-point-bending test. KHN (n = 6) was measured after 24 h dry storage and again after immersion in 100% ethanol solution for 24 h, to calculate PCL density. Data were analyzed by appropriate statistical analyses.

Results. The pre-heated composite showed better MA than the room-temperature groups. A higher number of gaps were observed in the room-temperature groups, irrespective of the energy density, mainly in the axial wall ($p < 0.05$). Composite pre-heating and energy density did not affect the DC, FS and PCL ($p > 0.05$).

Significance. Pre-heating the composite prior to light polymerization similar in a clinical situation did not alter the mechanical properties and monomer conversion of the composite, but provided enhanced composite adaptation to cavity walls.

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1. Introduction

The most common reasons for composite restoration replacement are fracture and secondary caries [1]. Therefore, techniques that promote higher conversion and mechanical properties without jeopardizing the marginal sealing are of

special interest. The first approach to this aim was made by the use of soft-start light polymerization methods [2] and more recently studies have focused on the influence of composite pre-heating on the composite properties [3–6].

It has been reported that increasing composite temperature up to 60 °C might enhance the conversion degree on the top and in 2 mm of the bottom surfaces [5].

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Composite pre-heating (60 °C) under an isothermal condition is capable of increasing monomer conversion, as molecular mobility is enhanced and collision frequency of reactive species is increased [3–6]. Composites with increased conversion are expected to be highly cross-linked and to have better mechanical properties [7]. As a consequence of this enhanced cross-linking; the free volume within the polymer network is reduced, as well as the solvent uptake and material degradation in the oral environment [8], although this issue is yet to be investigated.

Despite the aforementioned advantages, Lohbauer et al. [9] showed that composite pre-heating may cause detrimental effects on the restoration margin as it increases the polymerization shrinkage of the resin composite. To the extent of the authors' knowledge, no other study has so far addressed the effects of composite pre-heating on marginal adaptation of composite restorations, which deserve further investigation.

Furthermore, it is worth mentioning that the studies that have demonstrated optimization in monomer conversion [3–6] generally maintained the composite temperature constant, a situation that cannot be reproduced in a clinical situation. Once composite is pre-heated, there is a time delay between dispensing it from a syringe or compute, placing it into a preparation, contouring it, and subsequently light polymerizing it. It is estimated that when a composite is heated up to 60 °C and removed from the device, the temperature reduces 50% after 2 min and 90% after 5 min [10]. Therefore, it is clinically important to evaluate the influence of pre-heating under a non-isothermal condition, to simulate the real clinical scenario.

Therefore the aim of this study was to evaluate the effect of pre-heating on marginal adaptation, monomer conversion, flexural strength, microhardness, and polymer cross-linking of a resin composite under a non-isothermal condition for two energy densities (12 and 24 J/cm²). Two hypotheses were tested: (1) pre-heating increases composite monomer conversion, flexural strength, polymer cross-linking and marginal adaptation of composite to the cavity; (2) increased energy density increases composite monomer conversion, flexural strength and polymer cross-linking, and marginal adaptation of the composite to the cavity.

2. Materials and methods

For all experimental conditions, the nanofiller resin composite Filtek Z350 (3M/ESPE, St. Paul, MN, USA; batch number 6AJ; shade C2) was used and light polymerized at 600 mW/cm² for 20 s (energy density = 12 J/cm²) or 40 s (24 J/cm²) (Table 1), with a quartz-tungsten-halogen light polymerization unit (Optilux

501, SDS Kerr, Danbury, CT, USA). Irradiance was checked daily with the built-in radiometer and with a Model 100 Optilux Radiometer (SDS Kerr, Danbury, CT, USA).

Composite pre-heating was performed by a Calset™ device (AdDent Inc., Danbury, CT, USA) that elevates composite temperature to 68 °C. The mean time between removing composite from the device and light polymerization was approximately 40 s for all tests. The specimen preparation and testing was performed at controlled room-temperature (25 °C).

2.1. Marginal adaptation (MA)

Forty sound bovine incisors were stored in 0.5% chloramine solution for no longer than three months after extraction. Teeth were randomly assigned to one of four experimental groups (Table 1, n=10). Cylindrical diamond burs (ref #2094, KG Sorensen, Barueri, SP, Brazil) were used to prepare class V cavities (4 mm wide × 2 mm long × 2 mm deep) with enamel margins and axial wall in dentin. The internal walls of each cavity (C-factor = 3) were perpendicular to the top and bottom surfaces, with round angles as defined by the bur shape.

Preparations were acid etched (35% phosphoric acid, 3M/ESPE, St. Paul, MN, USA) for 15 s, washed and gently dried. The adhesive system Adper Single Bond 2 (3M/ESPE, St. Paul, MN, USA; batch number 6FT) was applied in two layers according to the manufacturer's directions and light polymerized for 10 s at 600 mW/cm². Resin composite either at room-temperature or pre-heated to 68 °C was placed in bulk and light polymerized for 20 or 40 s.

Restorations were then stored in distilled water at 37 °C for 7 days. Each specimen was sectioned in half through the center of the restoration with a diamond saw (Exttec-Blade XL 12235, Exttec Corp., Enfield, CT, USA) in a cutting machine (Labcut 1010 Exttec Corp., Enfield, CT, USA), resulting in two fragments with the adhesive interface exposed. Both fragments were embedded in epoxy resin and polished down using decreasing grit abrasive papers (600, 1200, 2400 and 4000, Buehler Ltd., Lake Bluff, IL, USA) and 0.5 μm diamond paste (Buehler Ltd., Lake Bluff, IL, USA) with a polishing cloth. Impressions of polished surfaces were taken with low viscosity vinyl polysiloxane material (Express, 3M/ESPE, St. Paul, MN, USA), which served as molds to fabricate epoxy resin replicas (Buhler Epoxicure Resin, Lake Bluff, IL, USA).

Replicas were platinum coated (MED 020, Bal-Tec, Liechtenstein) and the gaps in the interface were analyzed at 200× magnification in a scanning electron microscope (SEM) (Stereo Scam/LEO, Cambridge, United Kingdom). Adhesive interfaces were divided into seven sections (Fig. 1). Each sec-

Table 1 – Experimental groups according to the pre-polymerization temperature and energy density.

Group	Pre-polymerization composite temperature	Irradiance (mW/cm ²)	Irradiation time (s)	Energy density (J/cm ²)
RT40	Room-temperature	600	40	24.0
PH40	Pre-heated	600	40	24.0
RT20	Room-temperature	600	20	12.0
PH20	Pre-heated	600	20	12.0

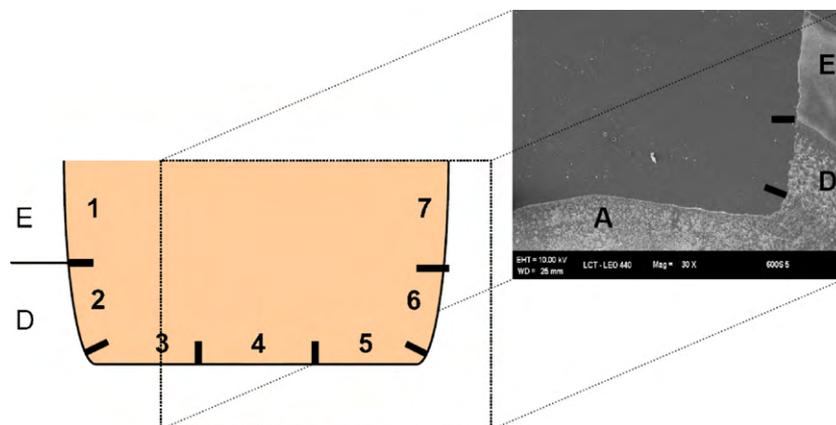


Fig. 1 – In the left figure, a schematic drawing shows a slice of a restoration. The slice of the restoration was divided into seven sections. Sections 1 and 7 are in lateral enamel, 2 and 6 in lateral dentin and 3–5 in axial wall. In the right figure, a scanning electron microscopy image partially shows the same restoration slice. Lateral enamel (E), lateral dentin (D) and axial wall (A).

tion received a score according to the presence of gap: 0 = no gaps observed; 1 = at least 1 gap observed. Higher SEM magnifications (300–1600 \times) were used to allow visualization of the gaps at the adhesive interface.

Two statistical analyses were performed. First, the scores given for the two tooth halves were averaged and a single score was then used per tooth. Data from the four experimental groups were analyzed by Kruskal–Wallis and Mann–Whitney's test ($\alpha=0.05$). Second, a mean of the scores attributed to the axial wall for the two tooth halves (sections 3–5) was averaged, as well as the proximal walls (sections 1, 2, 6 and 7), in order to compare the frequency of gaps in the proximal and axial walls. Data were analyzed by Wilcoxon's test for segment mean score comparisons and Friedman's test ($\alpha=0.05$).

2.2. Degree of conversion (DC)

Cylindrical specimens ($n=3$), 2 mm high and 5 mm in diameter were built-in a split Teflon mold positioned between mylar strips. Specimens were stored dry for 24 h at 37 °C and then submitted to degree of conversion (DC) analysis using FT-Raman spectroscopy (RFS 100/S, Bruker Analytische Meßtechnik, Karlsruhe, Germany), with an Nd-Yag laser. Both irradiated and non-irradiated surfaces of the composite were analyzed. Spectra were obtained by co-addition of 128 scans, at a resolution of 4 cm^{-1} . DC was obtained by standard baseline techniques. The percentage of unreacted carbon–carbon double bonds (% C=C) was determined from the ratio of absorbance intensities of aliphatic C=C (peak height at 1640 cm^{-1}) against internal standard before and after specimen polymerization. The aromatic carbon–carbon bond (peak height at 1610 cm^{-1}) absorbance was used as an internal standard. The degree of conversion (DC) was determined by subtracting the % C=C from 100%.

2.3. Flexural strength (FS)

For FS measurements, 10 specimens of each experimental condition were prepared using a stainless steel mold (10 mm \times 2 mm \times 1 mm), positioned over a polyester strip. After filling the mold to excess, the material surface was covered with a polyester strip and a glass slide and compressed under a 500 g load to extrude excess material. The specimens were light activated according to the protocols already described in Table 1. Specimen dimensions were measured using a digital caliper (Digimatic Caliper CD-6" OS, Mitutoyo, Japan). Samples were then stored in distilled water at 37 °C for 24 h.

The three-point bending test was carried out in a universal testing machine (Kratos Dinamômetro, São Paulo, Brazil), at a cross-head speed of 0.5 mm/min and 8 mm span between supports. FS was calculated with the following formula:

$$FS = \frac{3 \times L \times D}{2 \times W \times H^2}$$

where FS, flexural strength (MPa); L, failure load (N); D, distance between the supports (mm); W, specimen width (mm) and H, specimen height (mm).

2.4. Polymer cross-linking estimate (PCL)

With the same mold used for the DC test, cylindrical specimens (2 mm high and 5 mm in diameter; $n=6$) were built-up for the microhardness test. The mold was placed over a polyester strip, filled with the resin composite in bulk, covered with another mylar strip and then light polymerized. Specimens were stored dry at 37 °C for 24 h. After that, indentations were made in the irradiated surface with a Knoop indenter (HMV-2T, Shimadzu Corporation, Kyoto, Japan), using a 100 g load for 1 min. Five indentations were made in each specimen and these values were averaged for statistical purposes.

After the first KHN measurement, specimens were stored in 100% ethanol solution at 37 °C for 24 h and second KHN measurement was performed. The polymer cross-linking density

Table 2 – Gap frequency for each section of adhesive interface for all experimental groups.

Group	Enamel	Dentin	Lateral wall (sum of enamel + dentin)	Axial	Total	Gaps (%) per experimental group
RT40	3	9	12	27	39	55.7 A
PH40	3	12	15	18	33	47.1 B
RT20	5	7	12	29	41	58.6 A
PH20	4	6	10	26	36	51.4 B
Gaps (%) per segment	10.0	22.8	32.8 b	67.1 a	–	–

Groups with same capital or lower case are statistically similar (Tukey's test, $p > 0.05$).

(PCL) was estimated by the softening effect promoted by the ethanol, i.e., by the decrease in hardness.

The data from DC, FS and PCL were analyzed by two-way ANOVA (pre-polymerization temperature and energy density) and Tukey's test ($\alpha = 0.05$).

3. Results

3.1. Marginal adaptation

The overall marginal adaptation values of all groups are shown in Table 2 and Figs. 2 and 3. The cross-product interaction (pre-polymerization temperature and energy density) was not statistically significant, as well as the main factor energy density ($p > 0.05$). Only the main factor pre-polymerization temperature was statistically significant ($p = 0.001$). Pre-heated composite showed fewer gaps at the tooth-restoration interface than room-temperature composite. A significant difference was found when the axial wall was compared with the enamel and dentin segments ($p < 0.05$). A higher number of gaps were found in the axial wall (Table 2).

3.2. Degree of conversion (DC), flexural strength (FS) and polymer cross-linking estimates (PCL)

The overall degree of conversion, flexural strength and polymer cross-linking estimated values of all groups are shown in Table 3. For all tests, the cross-product interaction, pre-polymerization temperature vs. energy density, as well as the main factors were not statistically significant ($p > 0.05$). Room-temperature and pre-heated composites exhibited similar FS, DC and PCL, irrespective of the energy density ($p > 0.05$).

4. Discussion

This study showed that monomer conversion, FS and PCL were not significantly affected by composite pre-heating. This can be explained by the rapid drop in composite temperature during handling, also previously mentioned by Lovell et al. [4] and Daronch et al. [10]. It takes almost 2 min to complete the steps of removing the composite from the Calset device, full cavity filling and material accommodation. It is estimated that when a composite is heated up to 60 °C and removed from the device, its temperature drops around 35–40% after 40 s [10]. This was

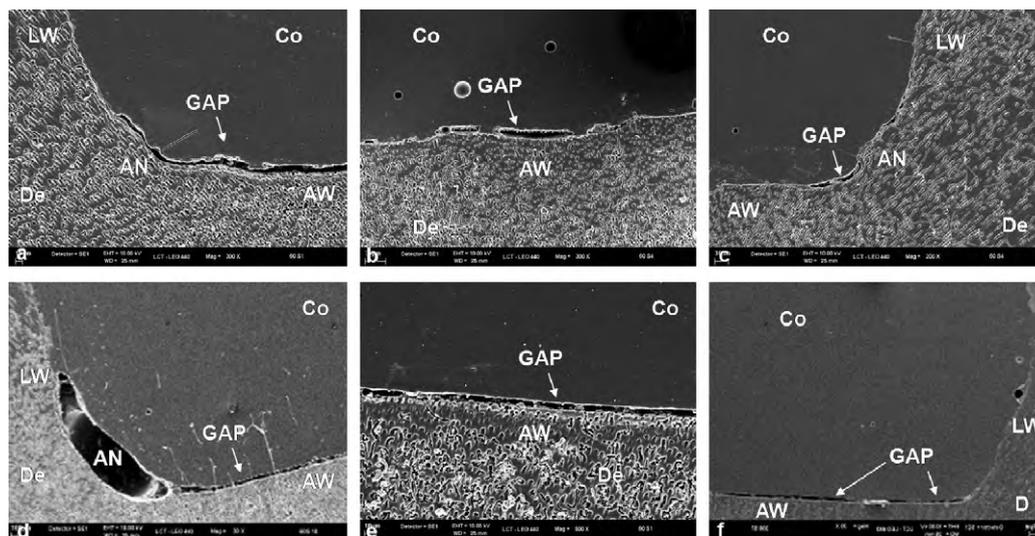


Fig. 2 – SEM images from epoxy resin replicas showing details of the adhesive interface for groups RT20 (a–c) and RT40 (d–f). Gaps in the axial wall can be seen in all pictures. In some cases, gaps can also be detected in the lateral wall (c) and in the cavity angle (a and d). Co = composite; De = dentin; AN = cavity angle; AW = axial wall; LW = proximal wall.

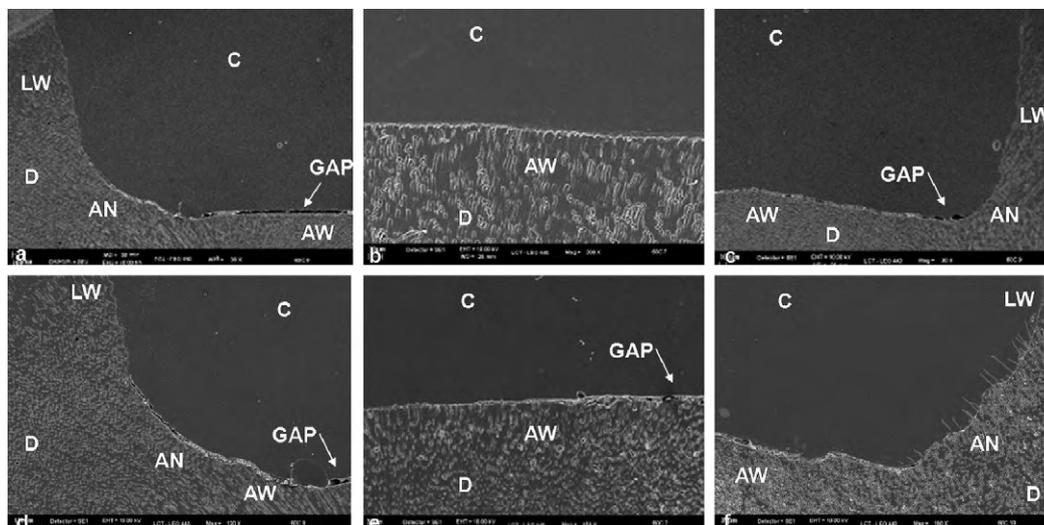


Fig. 3 – SEM images from epoxy resin replicas showing details of the adhesive interface for groups PH20 (a–c) and PH40 (d–f). Gaps can be seen in some pictures, usually in the axial wall (a, c–e). Co = composite; De = dentin; AN = cavity angle; AW = axial wall; LW = proximal wall.

the amount of time required to fill the cavities in this study. Therefore it is likely that by the time the composite is light activated, its temperature had reached a physiological level at which the temperature might not be sufficient to promote an increase in molecular mobility and consequent enhancement in the mechanical properties of the composite.

Moreover, it is worth mentioning that the degree of conversion and flexural strength were analyzed only 24 h after polymerization. It is well known that the degree of conversion of composites increases significantly after polymerization, and this fact could have masked the slight increase in DC that occurs for composites with pre-heating [10]. This was in fact observed by Lovell et al. [4]. The authors reported that the advantageous effect provided by composite pre-heating in terms of enhanced DC was much less pronounced after 24 h, when compared with immediate measurements. It is known that room-temperature composite can increase its conversion up to 10% [11,12] over time and this phenomenon is unlikely to occur with pre-heated composites, since a higher conversion is reached immediately. Thus a further increase in the DC is unlikely, as a higher restriction in later monomer mobility is observed [3,12] being responsible for the similarity between groups.

A previous study [5] demonstrated that pre-heated composite allows the duration of light exposure to be reduced, resulting in similar or better conversion than when the composite is irradiated for longer exposures at 22 °C. The aforementioned authors reported that a 5-s exposure (one-fourth of the time usually recommended) for a pre-heated composite (57 °C) resulted in a conversion rate (52–64%) higher than that observed after a 40-s exposure for room-temperature composite (48%). This may explain why the use of different energy densities (12 and 24 J/cm²) did not provide any enhancement in the monomer conversion and mechanical properties for pre-heated composites.

However, this fact does not explain why no significant difference was also observed for pre-heated composites light polymerized for different light exposures. The 40 s exposure time used in this study is twice the time recommended by the manufacturer. The conversion degree of the nanofilled composite used in this study, when light polymerized for 20 s at 600 mW/cm² (12 J/cm²) was higher than 60%, reducing the possibility of further increases in the conversion rate by increased duration of light polymerization. Nanofilled composite shows high translucency levels due to little light scattering effect [13]

Table 3 – Overall means for degree of conversion, flexural strength and polymer cross-linking estimation and the respective standard deviations (%) obtained in each experimental condition, as well as the statistical significance.

Group	Degree of conversion (%)	Flexural strength (MPa)	Polymer cross-linking estimation (%)
RT40	64.8 (3.0) A	181.1 (17.2) a	30.6 (4.3) ^a
PH40	60.9 (3.0) A	167.6 (14.6) a	30.0 (2.8) ^a
RT20	63.0 (2.7) A	179.3 (9.4) a	30.1 (3.0) ^a
PH20	65.1 (1.9) A	179.3 (17.9) a	31.7 (5.2) ^a

Groups with same capital, lower or superscript case are statistically similar (Tukey's test, $p > 0.05$).

which can favor the attainment of high conversion values. Thus, an energy density of 12J/cm² was shown to be sufficient to produce an adequate degree of conversion, flexural strength and PCL for Filtek Z350.

Unfortunately, the degree of conversion and other properties may vary from brand to brand and from shade to shade. Direct extrapolation of relationships observed in the present study to all other composites may not be valid, because of differences in filler composition and chemistries.

Interestingly, the previous expectation that composite pre-heating could worsen the marginal adaptation of composites to the cavity walls due to the increased conversion rates [5,12] and consequently increased polymerization shrinkage [9,14] was not confirmed by the results of the present investigation. This was also expected because resin composites exhibit a six to eight times greater thermal expansion than the surrounding tooth structures [15], polymerization shrinkage along with thermal contraction might create high interfacial stresses in pre-heated composites upon thermal equilibrium, with detrimental effects on marginal adaptation, integrity and seal [16].

After 24 h, the pre-heated groups showed better marginal adaptation than the room-temperature composites. This could be attributed to the reduction in the composite viscosity [17] and better adaptation to the cavity, especially in angled areas. This is an important clinical finding, as perfect sealing is essential to improve restoration longevity [1] and to prevent postoperative sensitivity [18]. It is likely that by the time the composite was placed within the cavity, its temperature was sufficiently high to allow better wetting of the cavity walls, although not high enough to improve mechanical properties and monomer conversion.

The intensity of the light used for light polymerization is greatest at the surface and decreases as it penetrates deeper into the composite [19]. Thus, the lower part of the mass, furthest from the light source, will reach the gel point more slowly than the top part, where the light intensity and polymerization rate are greater. Therefore, more stress relief by flow will occur in the deeper part of the composite [20], which might explain the worse marginal adaptation at the axial wall.

Moreover, it cannot be ruled out that bond strength in the deep dentin areas is usually lower [21], due to a reduced intertubular dentin area and increased dentinal tubule density [22]. As the angles of the cavity were included in the axial segment for data analysis, it is also possible that the higher number of gaps in axial area corresponds to areas of greater polymerization stress concentration [23] or difficult composite adaptation.

5. Conclusions

Under non-isothermal conditions (similar to a clinical situation) composite pre-heating to 68 °C did not improve the degree of conversion, flexural strength or polymer cross-linking, but yielded enhanced marginal adaptation. The marginal adaptation was significantly better in the axial wall when composite was pre-heated. For the composite studied an energy density of 12J/cm² seems to be sufficient to provide the composite with adequate mechanical properties.

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