



Influence of Pre-Heating Regular Resin Composites and Flowable Composites on Luting Ceramic Veneers with Different Thicknesses

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The aim of this study was to evaluate of pre-heating, filler contents and ceramic thickness on film thickness, microshear bond strength, degree of conversion and color change on ceramic veneers. Two experimental composites were prepared (Bis-GMA/UDMA/BisEMA/TEGDMA), with different amounts of filler (65% or 50%wt) simulating a conventional and a flowable composite. The flowable (F) was used at room temperature and, the conventional either at room temperature (C) or pre-heated (CPH). Disk-shaped ceramics with different thickness (0.4 mm, 0.8 mm, 1.5 mm) were prepared. The film thickness was evaluated according to the ISO 4049 (n=10). The microshear bond strength (n=10) was evaluated in enamel using tubing specimens light-cured through the ceramic veneer. The degree of conversion was evaluated using Raman spectroscopy. The color change of the ceramic restorations (n=10) was evaluated by spectrophotometry. The results were submitted to 2-way ANOVA and Tukey's post hoc test ($\alpha=5\%$). For the film thickness 1-way ANOVA was used ($\alpha=5\%$). The C presented the thicker film thickness; the CPH produced a similar film thickness in comparison to the F. All composites showed similar microshear bond strength. The degree of conversion of the F was higher than the C and CPH. The degree of conversion of the composites photo-activated through a 0.4 mm was higher than the composites photo-activated through thicker ceramics. The C showed the highest color change, while the CPH showed similar color change to the F. In conclusion, pre-heated conventional composites seem to be a potential alternative to lute ceramic veneers such as the flowable composites.

Key Words: film thickness, degree of conversion, model composite, experimental composite.

Introduction

It is a consensus among experts that healthy dental tissue should be protected from unnecessary wear (1-4). Since a worn out dental tissue will never be recovered, minimum wear is indispensable, and consequently, indirect restorations not necessarily need to be so thick. However, it does make the substrate color as well as the luting agent color being extremely important for the final esthetic appearance of the restoration (5). Another factor of extreme importance when talking about indirect restorations is the bond strength between the luting agent and the dental substrate and/or the restorative material.

Resin composites have become one of the most versatile materials used in Dentistry. In its usual indication, resin composites are used in direct restorations, allowing the clinicians to sculpt the dental shape and mimic optical characteristics of the dental structures. However, the resin composites can also be used to build-up indirect restorations to be cemented later, as well as in the fixation or cementation of these indirect restorations or others made of ceramic.

The composition of the resin composites is based on the triad organic matrix or resin blend (monomers), fillers (silica,

quartz or ceramic glass) and a photoinitiator to initiate the polymerization reaction (such as the camphorquinone, TPO, BAPO or others). Compared to the resin cements composition, the two major differences would be the type of monomers used in the resin blend and the amount of filler, giving to the resin cement lower viscosity to be more flow, but reducing the mechanical properties in comparison to the conventional composites. Thus, due to the great versatility of the composites and their superior mechanical properties, alternative techniques have been used to reduce the viscosity of the conventional composites so they could be used as a luting agent. The most known technique is called the thermoplastic technique, where the regular conventional composite becomes less viscous when heated due to increased agitation of the molecules, providing sufficient viscosity for the fixation of indirect restorations, similarly to the resin cements. Thus, it is expected that this heated composite could be used as a luting agent without losing its mechanical strength properties (6).

A conventional composite would not allow the correct placing of an indirect restoration, leading to a very thick film of material and, consequent marginal disadaptation. Then, the thermoplastic technique would reduce the viscosity of

composite by increasing its temperature (7) and making possible to correctly place an indirect restoration. It is not known, however, whether using a conventional composite for cementation would increase the bond strength between restorative material and substrate or if it would affect its mechanical and/or its rheological properties.

Moreover, there are no studies comparing the color changing of resin composites when they are heated and used as a fixation agent of ceramic veneers, as well as, the influence of the ceramic veneer thickness on the final color of these restorations over time. Thus, the aim of this study was to evaluate the factors that could affect color and bond strength of ceramic veneer restorations when using regular resin composites as the luting agent by evaluating the film thickness of model resin composites with different viscosities (flowable composite, conventional composite (control) and pre-heated conventional composite) and the degree of conversion, the bond strength and the color change of these composites when photoactivated through ceramic veneers with different thicknesses. For statically purpose, the conventional composite when non-heated was used as a control. The tested hypotheses were: 1) the filler content and the pre-heating will have influence on the film thickness; 2) the filler content and the pre-heating will have influence on the degree of conversion; 3) the filler content and the pre-heating will have influence on the bond strength to the enamel; 4) the filler content and the pre-heating will have influence on the color change of the restoration overtime; 5) the thickness of ceramic veneer will have influence on the degree of conversion, bond strength and color change of the ceramic restorations.

Material and Methods

Study Design

The experimental variables were film thickness (FT), μ -shear bond strength (μ SBS), degree of conversion (DC) and color change (CC). For the experimental variable FT, there was one experimental level: Experimental composites - (F) - Flowable, (C) - Conventional, and (CPH) Conventional Pre-Heated. The response variable was measured in mm and the association of levels consisted of 3 experimental groups ($n=10$). For μ SBS, DC and CC experimental variables, two experimental levels were considered: Experimental composites - F, C, and CPH; and Ceramic thickness - 0.4 mm, 0.8 mm, and 1.5 mm. The response variable for μ SBS was MPa ($n=10$). For DC was% ($n=5$) and for CC was ΔE_{00} ($n=10$). The association of the levels for these experimental variables constituted 9 experimental groups.

Experimental Resin Composites

The two resin composites were prepared with the same resin blend containing 29 wt% of Bis-GMA (Sigma

Aldrich, St. Louis, MO, USA), 32.5 wt% of UDMA (Sigma Aldrich), 32.5 wt% of Bis-EMA (Sigma Aldrich) and 6 wt% of TEGDMA (Sigma Aldrich). However, to this resin blend, different amounts of fillers were added: 65 wt% of filler for the conventional composite, in which 13 wt% was 0.05 μ m fumed silica (Nippon Aerosil Co. Ltd., Yokkaichi, Tokyo, Japan) and 52 wt% was 0.7 μ m BaBSiO₂ glass (Esstech Inc., Essington, PA, USA); or 50 wt% of filler for the flowable composite in which 10 wt% was 0.05 μ m fumed silica (Nippon Aerosil Co. Ltd., Yokkaichi, Tokyo, Japan) and 40 wt% was 0.7 μ m BaBSiO₂ glass (Esstech Inc., Essington, PA, USA). To all composites, 0.25 wt% of camphorquinone (Sigma Aldrich, St. Louis, MO, USA) and 0.50 wt% of ethyl-4-dimethylamino benzoate (Sigma Aldrich, St. Louis, MO, USA) were added to the resin blend as the photo-initiator system with 0.01 wt% of 2,6-bis(1,1-dimethylethyl)-4-methylphenol (Sigma Aldrich, St. Louis, MO, USA) as the photo-polymerization inhibitor.

The flowable resin composite (F) was used at room temperature and, the conventional resin composite was used either at room temperature (C) or pre-heated at 60°C (CPH). The CPH were always kept in syringes at 60°C until used. All experimental resin composites were stored in opaque plastic containers and protected from light exposure. All samples preparation and testing procedures were carried out at controlled room temperature (25 ± 2 °C) and humidity ($50 \pm 5\%$). The heating of the conventional composite was performed in a dry incubator (ThermoSmart, AstoriLab, Poncarale, BS, Italy) up to 60°C.

Ceramic Veneers Preparation

Ninety-three ceramic disc-shaped specimens with 7.5 mm in diameter (31 with 0.4 mm, 31 with 0.8 mm and 31 with 1.5 mm in thickness) were prepared using the IPS e.max Press ceramic system (Ivoclar Vivadent, Schaan, Liechtenstein), shade HT A1, following the manufacturer's instructions. These 31 ceramic discs-shaped simulating veneers restorations in each group were divided into 3 subgroups ($n=10$), according to the resin composite used (C, CPH and F) for the color change analysis. The remaining ceramic specimen was used during all photo-activation procedures, as shown in the Figure 1, as further described in each method. One surface of these discs was polished and the other etched with 10% hydrofluoric acid to simulate the clinical conditions.

Film Thickness (FT)

The film thickness evaluation ($n=10$) was performed according to ISO 4049:2009 (8) specification, where the thickness of 2 overlapping glass plates is measured four times before and after the insertion of the resin composite between the glass plates using a digital caliper, and the

film thickness calculated as the difference in between the measurements. The temperature of the glass plates was standardized at 37 °C to simulate the clinical conditions. A standardized volume of each composite (0.10 mL) was placed between the two glass plates and a standard load of 150 N was applied on the top plate for 180 s. The volume of the composites used was measured according to the syringe's volume markings in the barrel. The photoactivation was performed through the glass plate for 20 s using a LED with 1,200 mW/cm² of irradiance (Bluephase G2, Ivoclar Vivadent, Schaan, Liechtenstein). The difference between the average of the final and initial measurements was considered the film thickness achieved by the resin composite.

Microshear Bond Strength (μ sbs)

Ninety enamel blocks (10x10 mm) from bovine incisors were embedded in acrylic resin (Clássico, Sao Paulo, SP, Brazil) into polyvinyl chloride tubes and wet-polished with 400- and 600-grit silicon carbide abrasive papers (Buehler, Lake Buff, IL, USA) to obtain flat enamel surfaces. The embedded enamel specimens were divided in 9 groups (n=10), according the resin composite and ceramic veneer thickness used. For all groups, the enamel surface was etched with 35% phosphoric acid gel (3M/ESPE, St. Paul, MN, USA), rinsed, dried and, then one coat of Scotchbond Multipurpose adhesive (3M/ESPE) was applied following the manufacturer's instructions. Before the light-activation, three translucent Tygon tubings (0.9 mm internal diameter x 0.5 mm in height) were positioned onto the enamel surface

of block specimen and used as molds (Fig. 1-A), and then, light-cured for 10s. Then, the respective experimental resin composite was carefully inserted with a snap-fit syringe and 20-gauge needle tubes (Centrix Corp, Shelton, CT, USA) into the Tygon tubes lumen. Only three Tygon tubings were filled each time for the CPH group to avoid temperature drop. For conventional composite it was necessary to use a root canal plugger (Odous de Deus, Belo Horizonte, MG, Brazil) to push the material into the tubes lumen. After filling all three Tygon tubing molds, the experimental resin composites were light-cured for 20 s through the one of the ceramic veneer discs (having 0.4, 0.8 or 1.5 mm in thickness) that was fixed onto the output region of the curing light tip (as shown in Fig. 1A) in order to be stabilized in the same position when curing the tubing specimens (as shown in Fig. 1B). For this, the curing light was clamped to a x-y stage (Edmund Optics, Barrington, NJ, USA) in order to be positioned right on top the tubing samples in the same position every time.

The specimens were stored in distilled water for 24 h at 37 °C and the three Tygon tubes of each specimen were removed using a scalpel blade. A Stereomicroscope (model SMZ-1B, Nikon, Japan) at 30x magnification was used to confirm that none of the cylinders presented defects or flaws at the bonding area. The PVC tube was positioned in a μ SBS device and properly fixed to a mechanical testing machine (Instron, model 4411, Canton, MA, USA). Then, a thin steel wire (0.2 mm in diameter) was looped around the base of one of each cylinder of each time and aligned with the bonding interface (Fig. 1C). Each cylinder was

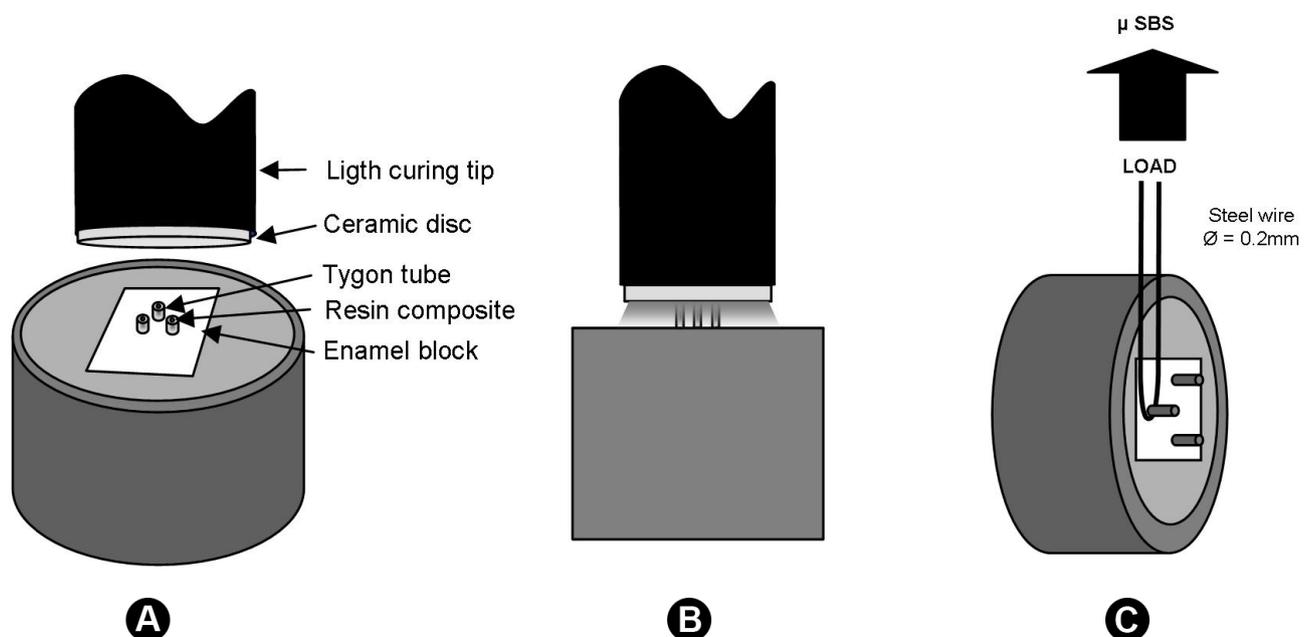


Figure 1. A: Schematic illustration of specimen's preparation and; B: Photoactivation through the ceramic disc; C: Steel wire looped around the resin composite cylinder for the μ SBS test.

submitted to a crosshead speed of 0.5 mm/min until failure. The mean of three cylinders of each specimen was considered as the mean of each specimen (n=10) for the statistical analysis. The debonded interfaces were examined under stereomicroscope (model SMZ-1B, Nikon) at 30× magnification and the failures were classified as: adhesive, cohesive within enamel, cohesive within resin and mixed (enamel/adhesive/resin).

Degree of Conversion (DC)

For the degree of conversion (DC) analysis of the experimental resin composites, 5 specimens per group with 6 mm in diameter and 1 mm in thickness were made in rubber molds and photo-activated for 20 s. This light-curing was done using a ceramic disc with each thickness (0.4, 0.8 and 1.5 mm) fixed onto the output region of the curing light tip in order to be stabilized in the same position using a x-y stage when curing the specimens, as previously described. After polymerization, the specimens were removed from the molds and dry stored in light-proof containers at 37 °C, for 72 h. DC was measured on the top surface of each specimen using FT-Raman spectroscopy (XploRA, Horiba, Kyoto, Japan) The absorption spectra of non-polymerized and polymerized composites were obtained from the region between 4000 and 650 cm⁻¹ with 32 scans at 4 cm⁻¹. The aliphatic carbon-carbon double-bond absorbance peak intensity (located at 1638 cm⁻¹) and that of the aromatic (C-C) (located at 1608 cm⁻¹; reference peak) were collected. The DC was calculated using the following equation: DC=100×[1-(R polymerized/R non-polymerized)], where R represents the ratio between the absorbance peak at 1638 cm⁻¹ and 1608 cm⁻¹.

Color Change (CC)

To measure color change, 10 specimens from each group were made by placing 0.05 mL of each resin on a polyester strip and pressing one of the ceramic veneers with the treated face facing the cement with 150 N and, then removing the excess and photoactivating through the ceramic for 20 s. CIELab co-ordinates (L, a, b) were recorded for each experimental composite pre- and immediately post- UV-light aging using a spectrophotometer (Vita EasyShade Advanced, VITA Zahnfabrik, Bad Säckingen, Baden-Württemberg, Germany) with a D65 illuminant. A white (L=14.9; a=2.1; b=9.0) paper was used as background for the color analysis. After the initial color measurements, all samples were UV-light aged for 120 h using only the UV-B cycle exposure at 37 °C (9) to simulate 10 years in clinical conditions (10). To standardize the UV-light exposure, the specimens were placed at 10 cm distance from the UV-light (UV-B 313, Equilam, Diadema, SP, Brazil).

To evaluate color change, ΔE_{00} of each specimen was

calculated using the following CIEDE2000 formula (11):

$$\Delta E_{00} = \left[\left(\frac{\Delta L}{k_{LS}S_L} \right)^2 + \left(\frac{\Delta C}{k_{CS}S_C} \right)^2 + \left(\frac{\Delta H}{k_{HS}S_H} \right)^2 + R_T \left(\frac{\Delta C}{k_{CS}S_C} \right) \left(\frac{\Delta H}{k_{HS}S_H} \right) \right]^{0.5}$$

where ΔL , ΔC and ΔH are the differences in lightness, chroma and hue, and R_T is a function (the so-called rotation function) that accounts for the interaction between chroma and hue differences in the blue region. The weighting functions, S_L , S_C , and S_H are used to adjust the total color difference for variation in the location of the color difference pair in the L, a, and b coordinates. The parametric factors k_L , k_C , and k_H , are correction terms for the experimental conditions, which were set to 1.

Statistical Analysis

Power analysis was conducted to determine sample size for each experiment to provide a power of at least 0.8 at a significance level of 0.5 ($\beta=0.2$). The FT data for each experimental resin composite were analyzed by one-way ANOVA and Tukey's pos-hoc test for pairwise comparisons. The factor considered was composite in three levels (C, CPH and F). The DC, μ SBS and CC data were analyzed by two-way ANOVA and Tukey's test for multiple comparisons. The two factors analyzed were composite in three levels (C, CPH, and F) and the ceramic thickness in three levels (0.4 mm, 0.8 mm and 1.5 mm).

Results

Film Thickness

The one-way ANOVA showed significant effect of the experimental resin composites on the film thickness ($p=0.00163$). The results for the Tukey's test and the means of film thickness are shown in Table 1. The conventional resin composite presented a thicker film in comparison to the flowable composite. However, there was no difference between the pre-heated conventional composite and the flowable composite.

Microshear Bond Strength

Table 2 shows the microshear bond strength (μ SBS) means and standard deviations for all experimental resin

Table 1. Film thickness (mm \pm standard deviation) of the model resin composites

Composite	Thickness (mm)
C	0.056 \pm 0.009 a
CPH	0.053 \pm 0.007 ab
F	0.047 \pm 0.006 b

Means followed by same small letter in the column are not statistically different at 5%, by Tukey's test.

composites photoactivated through the ceramic veneers with the different thicknesses. The two-way ANOVA demonstrated that composite ($p=0.24560$) and ceramic thickness ($p=0.09855$) factors were not significant, as well as the interaction between these factors ($p=0.97403$). The thickness of the ceramic discs did not influence on the uSBS means, regardless of the composite used. The uSBS means were similar to all resin composites tested, regardless of ceramic veneer thickness.

Figure 2 shows that for all groups with flowable resin composites (0.4 mm, 0.8 mm and 1.5 mm thickness ceramic veneers) the mixed failures were prevalent. For conventional composites groups (heated or not and with 0.4 mm, 0.8 mm or 1.5 mm thickness ceramic veneers), there were similar percentages of adhesive and mixed failures.

Degree of Conversion

Table 3 shows the degree of conversion (%) means and standard deviations for all experimental resin composites photoactivated through ceramic veneers with different

thicknesses. The two-way ANOVA demonstrated that composite ($p=0.00075$) and ceramic thickness ($p=0.00019$) factors were significant, but not the interaction between them ($p=0.26510$). The experimental resin composites photo-activated through a 0.4 mm ceramic veneers showed higher degree of conversion in comparison to resin cements photoactivated through a 1.5 mm ceramic veneer.

Color Change

Table 4 shows the color change (ΔE_{00}) means and standard deviations for all experimental resin composites photoactivated through ceramic veneers with different thicknesses. The two-way ANOVA demonstrated that composite ($p=0.00001$) and ceramic thickness ($p=0.02642$) factors were significant, but not the interaction between them ($p=0.63675$). The experimental resin composites photo-activated through a 0.4 mm ceramic veneer showed the highest ΔE_{00} mean and differed from the composites photo-activated through a 1.5 mm ceramic veneer. The resin composites photo-activated through a 0.8mm

ceramic veneer presented intermediate ΔE_{00} mean and did not differ from the composites photo-activated through the other ceramic thicknesses. The conventional composite presented the highest ΔE_{00} and differed from the flowable composite and from the pre-heated conventional composite, which did not differ from each other.

Table 2. Microshear bond strength means (MPa \pm standard deviation) for the model resin composites light-cured through the ceramic veneers with different thicknesses

Composite	Ceramic veneer thickness			Pooling mean
	0.4 mm	0.8 mm	1.5 mm	
F	35.64 \pm 6.88	32.92 \pm 7.39	32.01 \pm 8.40	33.52 \pm 7.48 a
C	37.05 \pm 11.24	34.76 \pm 11.63	31.64 \pm 10.89	34.48 \pm 11.09 a
CPH	41.28 \pm 5.80	35.81 \pm 9.47	35.09 \pm 8.22	37.39 \pm 8.19 a
Pooling mean	37.99 \pm 8.38 A	34.50 \pm 9.39 A	32.91 \pm 9.06 A	-

Means followed by same small letter in the column and capital letter in row, are not statistically different at 5%, by Tukey's test.

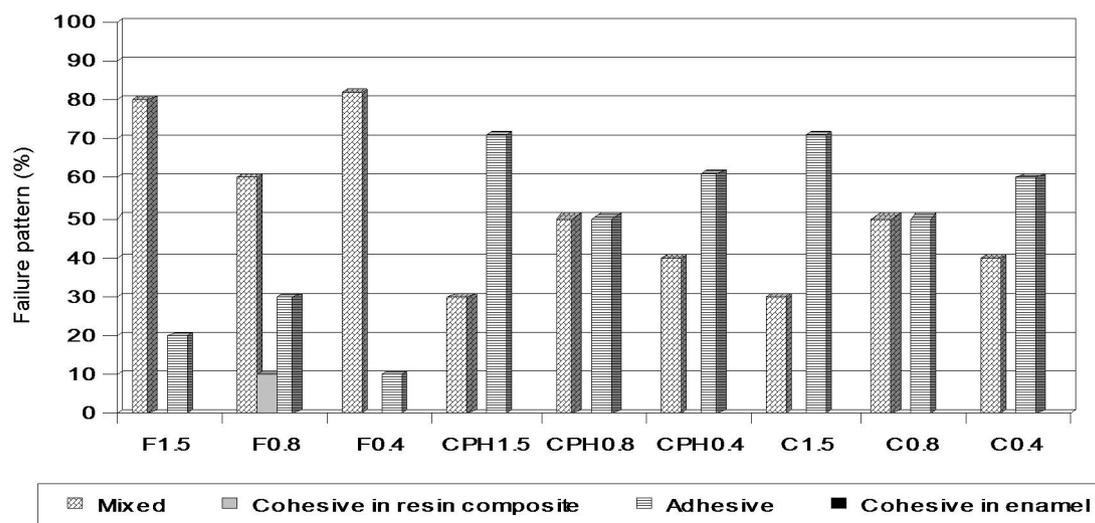


Figure 2. Failure pattern analysis of the debonded specimens (%).

have influence on the film thickness was accepted. First, the film thickness formed by the model composites was directly influenced by the filler content. As observed in the results the flowable composite showed a thinner film in comparison to the conventional composite. Second, the pre-heating of the conventional composite might not have reduced the film thickness enough to be statistically different from the conventional composite itself without using the pre-heating technique, as already shown in previous studies (12,13). However, it did reduce the film thickness enough to be statistically similar to the film thickness formed by the flowable composite (Table 1). The film thickness is an important factor when luting indirect restorations. Thicker film thicknesses may lead to the marginal misfit of these indirect restorations. Also, due to the greater amount of resin material to be polymerized, a greater volumetric shrinkage would also be expected, increasing the susceptibility to failure (14). The thinner film thickness formed by the conventional composite that was pre-heated probably occurs due to the increased agitation of molecules and mass plasticization of unpolymerized material, which reduces the viscosity (15). And, as expected, since the model composites had the same polymer matrix, the higher amount of fillers in the conventional composite increased the viscosity and, consequently, increased the film thickness in comparison

to the flowable composite. Of course, this evaluation was only possible because experimental composites varying only the amount of filler content were used in this study.

Regarding the degree of conversion analysis, the second tested hypothesis that the filler content and the pre-heating would have influence on the degree of conversion was accepted. There was a statistical difference between the conventional composites with highest filler content and the flowable composite with lowest filler content. The degree of conversion of the flowable composite was higher than the degree of conversion of the conventional composite (either pre-heated or not). The higher content of filler particles in the conventional composites may have reflected more of the incident light, reducing light-transmittance through the resin material and, consequently, reducing the degree of conversion (16). In addition, the higher molecular mobility in the early stages of the polymerization reaction in the flowable composite may have also contributed to the higher conversion of the polymer network into formation in comparison to the conventional composite. However, the pre-heating did not influence on statistical difference in the degree of conversion of the conventional composite. However, it is possible that other studies find an increase in immediate degree of conversion of the conventional composite by pre-heating it before curing, because when the composite is preheated, there

is a greater shock of molecules coming from the greater molecular agitation, accelerating the reaction (17). However, the differences between the degrees of conversion measured immediately after photopolymerization and over time tend to fall and potentially become null after 24 h (18).

On the other hand, the bond strength of the composites to the enamel was not affected neither by the amount of filler nor by the pre-heating of the conventional composite. Then, the third tested hypothesis that the filler content and the pre-heating would have influence on the bond strength to the enamel was rejected. However, other studies did find differences in the bond strength between pre-heated and non-heated conventional composites and dentin (19). In this study, it was chosen to use enamel as a substrate because it is more homogeneous than dentin and also because it allows to test the bond strength after the passage of light through the ceramic veneer and resin material.

Table 3. Degree of conversion means (% \pm standard deviation) for the model resin composites light-cured through the ceramic veneers with different thicknesses

	0.4 mm	0.8 mm	1.5 mm	Pooling mean
F	82.99 \pm 2.22	80.43 \pm 1.60	77.88 \pm 2.98	80.43 \pm 3.06 a
C	78.66 \pm 1.11	78.13 \pm 1.67	76.97 \pm 3.16	77.92 \pm 2.13 b
CPH	80.11 \pm 1.44	76.23 \pm 1.24	75.55 \pm 2.31	77.29 \pm 2.62 b
Pooling mean	80.59 \pm 2.41 A	78.26 \pm 0.58 AB	76.80 \pm 2.81 B	-

Means followed by same small letter in the column and capital letter in row, are not statistically different at 5%, by Tukey's test.

Table 4. Color change means (ΔE_{00} \pm standard deviation) for the model resin composites light-cured through the ceramic veneers with different thicknesses

Composite	Ceramic veneer thickness			Pooling mean
	0.4 mm	0.8 mm	1.5 mm	
F	1.19 \pm 0.80	1.16 \pm 0.11	1.13 \pm 0.15	1.16 \pm 0.46 b
C	2.33 \pm 0.70	1.98 \pm 0.75	1.82 \pm 0.12	2.04 \pm 0.61 a
CPH	1.80 \pm 0.71	1.46 \pm 0.43	1.28 \pm 0.15	1.51 \pm 0.52 b
Pooling mean	1.77 \pm 0.85 A	1.53 \pm 0.59 AB	1.41 \pm 0.33 B	-

Means followed by same small letter in the column and capital letter in row, are not statistically different at 5%, by Tukey's test.

Although is difficult to compare bond strength between different substrates such as enamel, dentin, and ceramic, it is important to point out that regardless no statistical difference was found for the bond strength between the composites and the enamel, most of the failures were either mixed failures between the composite and the adhesive, as for the flowable composite, or adhesive, as for the conventional composites. This shows that the viscosity of the composite does play a role in imbrication and bond strength in between the composites and the dental substrate. However, heating did not seem to influence in this, as pre-heated and non-heated conventional composites presented similar failure pattern, as observed in Figure 2.

The fourth tested hypothesis that the filler content and the pre-heating would have influence in the color change of the restoration over time was accepted. As observed in the results, both pre-heating and filler content did in fact affected the color change over time. The overall color change induced by the UV-light aging used in the study occurs due to the absorption of the UV-light by the tertiary amines (co-initiator) that, thus, create higher energy states. Then, these amines with a high degree of excitation react with oxygen, other aromatic groups or small organic molecules, which can be incorporated during the manipulation of the material (9,20). From these reactions, color centers are formed, also called chromophores, which increase the absorption of visible light, particularly in the blue region of the electromagnetic spectrum, causing the yellowing of the material over time (9,21). However, as observed in the results, the conventional composite showed the highest color change values, while the pre-heated conventional composite showed similar color change in comparison to the flowable composite. As showed before, the flowable composite showed a thinner film in comparison to the conventional composite. And, pre-heating the conventional composite did reduce the film thickness enough to be statistically similar to the film thickness formed by the flowable composite. Thus, it would be expected that as all the composites had the same amount of photoinitiator and co-initiator (tertiary amine), as the amount of the resin material is reduced due to the formation of a thin layer of film during the cementation, the amount of the tertiary amines able to react and cause discoloration over time is also reduced. Thus, explaining the results found for color change overtime for the conventional composites when pre-heated or not.

On the other hand, the amount of filler did also influence the color change of the composites over time. The conventional composite with highest filler content presented higher color change in comparison to the flowable composite with lowest filler content. Studies

published by De Oliveira et al. (16) and Fróes-Salgado et al. (22) also observed that the higher the amount of filler, the greater the color change of the composites. The greater the amount of filler in a composite, the greater the light reflection and the greater its attenuation during photoactivation (16,22). As observed in this study, the degree of conversion in fact decreased with the greater amount of filler content. Accordingly, the greater the number of unreacted monomers and amines, which will be degraded after the UV-light aging, greater the color change after aging.

The fifth tested hypothesis that the thickness of the ceramic veneer would have influence on the degree of conversion, bond strength and color change of the ceramic restorations was rejected. The results showed that the thickness of the ceramic veneer did have influenced on the degree of conversion and on the color change but did not have influenced on the bond strength to the enamel. The degree of conversion of the composites photo-activated through the thinner ceramic veneer with 0.4 mm was higher than the degree of conversion of the composites photo-activated through the thicker ceramic with 1.5 mm. Of course, that with the progressive attenuation of the light through the ceramic as thicker was the veneer, the less light being transmitted to the luting agent below the ceramic would directly influence on the degree of conversion of this photo-activated resin material. However, the decrease in the degree of conversion values does not always directly affect the bond strength values. In this study, for example, all composites presented similar microshear bond strength, regardless the ceramic veneer thickness or the degree of conversion achieved. It is noteworthy that not only the degree of conversion of the resin material plays a role in the physical and mechanical properties of resin materials, but also the type of polymer that is being formed. Thus, it is possible that the thickness of the ceramic veneer directly has influence on the degree of conversion, but not necessary decrease the overall bond strength values. Furthermore, as previously mentioned, it would be expected that as the amount of the resin material is reduced due to the formation of a thin layer of film during the cementation, the amount of the tertiary amines able to react and cause discoloration over time is also reduced, explaining the results found for color change overtime for the flowable and pre-heated conventional composites in comparison to the non-heated conventional composite.

In view of all the hypotheses, it seems evident that there are advantages and disadvantages of using the thermoplastic technique. For clinical implications, it is up to the clinic dentist to choose the way to perform his rehabilitations taking into account factors not applicable in laboratory tests such as technical skills, clinical experience

and availability of materials and equipment to be used. Thus, within the limitations of this study, it is possible to conclude that the pre-heated conventional composites seem to be a potential alternative to lute ceramic veneers such as the flowable composites.

Resumo

O objetivo neste estudo foi avaliar a influência do pré-aquecimento, conteúdo de carga e espessura da cerâmica na espessura da película, resistência de união ao microcisolamento, grau de conversão e alteração de cor na cimentação de cerâmicas. Dois compósitos experimentais foram preparados (Bis-GMA / UDMA / BisEMA / TEGDMA), com diferentes quantidades de carga (65% ou 50% em peso), simulando um compósito convencional e um fluido. O fluido (F) foi usado à temperatura ambiente e o convencional, à temperatura ambiente (C) ou pré-aquecido (CPH). Foram preparados discos de cerâmica com diferentes espessuras (0,4mm, 0,8mm, 1,5mm). A espessura da película foi avaliada de acordo com a ISO 4049 (n=10). A resistência de união ao microcisolamento (n=10) foi avaliada em esmalte utilizando espécimes fotopolimerizados através da cerâmica. O grau de conversão foi avaliado usando espectroscopia Raman. A alteração de cor das restaurações cerâmicas (n=10) foi avaliada por espectrofotometria. Os resultados foram submetidos à ANOVA 2-fatores e ao teste *post hoc* de Tukey ($\alpha=5\%$). Para espessura de película foi utilizada a ANOVA 1-fator ($\alpha=5\%$). O grupo C apresentou maior espessura de película; o CPH produziu uma espessura semelhante ao F. Todos os compósitos mostraram resistência de união ao microcisolamento similares. O grau de conversão do F foi maior que C e CPH. O grau de conversão dos compósitos fotoativados através 0,4 mm foi maior que dos compósitos fotoativados através de cerâmicas mais espessas. O grupo C apresentou a maior mudança de cor, enquanto o CPH apresentou mudança de cor semelhante ao F. Em conclusão, compósitos convencionais pré-aquecidos assim como compósitos fluidos, parecem ser uma alternativa em potencial para cimentar laminados cerâmicos.

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References

- Gaengler P, Hoyer I, Montag R, Gaebler P. Micromorphological evaluation of posterior composite restorations – a 10-year report. *J Oral Rehabil* 2004;31:991-1000.
- Ilie N, Hilton TJ, Heintze SD, Hickel R, Watts DC, Silikas N, et al. Academy of Dental Materials guidance-Resin composites: Part I-Mechanical properties. *Dent Mater* 2017;33:880-894.
- Wakiaga J, Brunton P, Silikas N, Glenn AM. Direct versus indirect veneer restorations for intrinsic dental stains. *Cochrane database Syst Rev* 2004;CD004347.
- Larson TD. 25 years of veneering: what have we learned? *Northwest Dent* 2003;82:35-39.
- Azer SS, Rosenstiel SF, Seghi RR, Johnston WM. Effect of substrate shades on the color of ceramic laminate veneers. *J Prosthet Dent* 2011;106:179-183.

- Magne P, Paranhos MPG, Burnett LH, Magne M, Belser UC. Fatigue resistance and failure mode of novel-design anterior single-tooth implant restorations: influence of material selection for type III veneers bonded to zirconia abutments. *Clin Oral Implants Res* 2011;22:195-200.
- Lucey S, Lynch CD, Ray NJ, Burke FM, Hannigan A. Effect of pre-heating on the viscosity and microhardness of a resin composite. *J Oral Rehabil* 2010;37:278-282.
- International Organization for Standardization. ISO 4049:2009(E). Dentistry – polymer-based filling, restorative and luting materials. Switzerland: International Organization for Standardization; fourth edition, 2009.
- de Oliveira DC, Ayres AP, Rocha MG, Giannini M, Puppini Rontani RM, Ferracane JL, et al. Effect of different in vitro aging methods on color stability of a dental resin-based composite using CIELAB and CIEDE2000 color-difference formulas. *J Esthet Restor Dent* 2015;27:322-330.
- Heydecke G, Zhang F, Razzoog ME. In vitro color stability of double-layer veneers after accelerated aging. *J Prosthet Dent* 2001;85:551-557.
- Sharma G, Wu WC, Daa EN. The CIEDE2000 color-difference formula: Implementation notes, supplementary test data, and mathematical observations. *Color Res Appl* 2005;30:21-30.
- Blalock JS, Holmes RG, Rueggeberg FA. Effect of temperature on unpolymerized composite resin film thickness. *J Prosthet Dent* 2006;96:424-432.
- Cantoro A, Goracci C, Papacchini F, Mazzitelli C, Fadda GM, Ferrari M. Effect of pre-cure temperature on bonding potential of self-etch and self-adhesive resin cements. *Dent Mater* 2008;24:577-583.
- Sampaio CS, Barbosa JM, Cáceres E, Rigo LC, Coelho PG, Bonfante EA, et al. Volumetric shrinkage and film thickness of cementation materials for veneers: An in vitro 3D microcomputed tomography analysis. *J Prosthet Dent* 2017;117:784-791.
- Schneider LFJ, Cavalcante LM, Consani S, Ferracane JL. Effect of co-initiator ratio on the polymer properties of experimental resin composites formulated with camphorquinone and phenylpropanedione. *Dent Mater* 2009;25:369-375.
- de Oliveira DCRS, de Menezes LR, Gatti A, Correr Sobrinho L, Ferracane JL, Sinhoreti MAC. Effect of nanofiller loading on cure efficiency and potential color change of model composites. *J Esthet Restor Dent* 2016;28:171-177.
- Daronch M, Rueggeberg FA, De Goes MF. Monomer conversion of pre-heated composite. *J Dent Res* 2005;84:663-667.
- Lohbauer U, Zinelis S, Rahiotis C, Petschelt A, Eliades G. The effect of resin composite pre-heating on monomer conversion and polymerization shrinkage. *Dent Mater* 2009;25:514-519.
- Demirbuga S, Ucar FI, Cayabatmaz M, Zorba YO, Cantekin K, Topçuoğlu HS, et al. Microshear bond strength of preheated silorane- and methacrylate-based composite resins to dentin. *Scanning* 2016;38:63-69.
- Stansbury JW. Curing dental resins and composites by photopolymerization. *J Esthet Dent* 2000;12:300-308.
- Cheung KC, Darvell BW. Sintering of dental porcelain: effect of time and temperature on appearance and porosity. *Dent Mater* 2002;18:163-173.
- Frões-Salgado NR, Silva LM, Kawano Y, Francci C, Reis A, Loguercio AD. Composite pre-heating: Effects on marginal adaptation, degree of conversion and mechanical properties. *Dent Mater* 2010;26:908-914.

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