Effect of pre-heated dual-cured resin cements on the bond strength of indirect restorations to dentin

Alexandre Morais[a]
Alline Rachid Abreu dos Santos[a]
Marcelo Giannini[b]
Andre Figueiredo Reis[a]
José Augusto Rodrigues[a]
César Augusto Galvão Arrais[a]

[a] Department of Restorative Dentistry, Dental Research Division, Guarulhos University – UnG, Guarulhos, SP, Brazil.

[b] Department of Operative Dentistry, University of Campinas, Piracicaba School of Dentistry, Piracicaba, SP, Brazil.

Abstract: This study evaluated the effects of resin luting agents (LA) polymerized using increased temperature on the *in vitro* microtensile bond strength (µTBS) of indirect restorations to dentin. The occlusal dentin surfaces of 40 human third molars were exposed and flattened. The teeth were assigned to 8 groups (n = 5) according to the LA temperature (25°C or 50°C), curing mode (dual- or self-curing mode), and product (Excite DSC/Variolink II [VII] and XP Bond/Calibra [Cal]). The bonding agents were applied to the dentin surfaces according to manufacturers' instructions. For preheated groups, the LAs were heated to 50°C, subsequently mixed on a heated stirrer surface, and applied to the previously heated pre-polymerized resin discs (2 mm thickness, TPH-Spectrum). The discs were bonded to the dentin surfaces, and the LAs were either exposed to a curing light according to manufacturers' instructions or allowed to self-cure. Specimens were stored in relative humidity at 37°C for 7 days. Specimens were mesio-distally and bucco-lingually sectioned to obtain multiple bonded beams with a 1-mm^2 cross-sectional area for µTBS testing. Data (MPa) were analyzed by 2-way ANOVA and Tukey's post hoc test (α = 5%) for each product. Specimen failure patterns were analyzed using a scanning electron microscope. VII groups showed higher µTBS at 50°C than at 25°C regardless of curing mode (p = 0.05). Cal groups showed similar µTBS at 25°C and 50°C in all activation modes. The use of some dual-polymerizing LAs at 50°C may improve the µTBS of indirect restorations to dentin.

Descriptors: Resin Cements; Temperature; Dental Bonding.

Introduction

Dual-polymerizing resin luting agents (LAs) are widely used to bond esthetic indirect restorations because of their low solubility, low viscosity, clinically acceptable film thickness, better mechanical properties than conventional cements, ability to bond tooth to restorative material when used with bonding agents, and lower microleakage compared to other luting materials.[1-3] In addition to the presence of photoiniators in their composition, self-curing components are added to ensure an optimal polymerization even when activating light is severely attenuated due to the presence of an indirect restoration interposed between LA and light-curing unit tip.[4-8] However, these components are not as effective as the dual-curing mode to provide an optimal polymerization.[4,5,7,9,10] Such

a low effectiveness is related to the slow activation and propagation rates of polymerization observed in the self-curing mechanism.[11]

Composite resin preheating to 60°C prior to resin polymerization decreases the resin viscosity and allows for an increased free radical mobility.[12-15] Consequently, these materials achieve higher monomer conversion at high temperature than when they are achieved at room temperature.[12,13,16-18] When monomer conversion is closely related to the polymer mechanical properties,[19-21] improved bond strength of indirect restorations to dentin would be expected even when LA polymerization relies solely on self-curing components. Although concerns about the possible effects of temperature on pulp may arise, previous findings have demonstrated that any increase in composite resin temperature to 54°C and 60°C does not significantly change the intrapulpal temperatures.[18] However, few studies have evaluated the effects of preheating resin LAs on the bond strength of indirect restorations to dentin.[22,23]

The aim of this study was to evaluate the effects of preheating dual-polymerizing LAs on the bond strength of indirect restorations to dentin. The research hypothesis was that preheated LAs provide greater bond strength to dentin than cements used at room temperature regardless of activating mode.

Methodology
Indirect restorative procedures

Forty freshly extracted, erupted human third molars were used. The research protocol was approved by the Human Assurance Committee of Guarulhos University (90/09). Teeth were stored in saturated thymol (Symrise GmbH & Co, Holzminden, Germany) at 5°C for no longer than 3 months and were sectioned perpendicular to the long axis using double-face diamond discs (7020; KG Sorensen, Cotia, Brazil) to expose middle-depth occlusal dentin surface. The dentin surfaces were wet ground (Arotec, Cotia, Brazil) with 600-grit SiC papers (Carborundum; Saint-Gobain Abrasivos, Guarulhos, Brazil) to create a flat surface with a standard smear layer.

Forty pre-cured resin composite discs (2-mm thickness and 10-mm diameter; A2 shade, TPH Spectrum; Dentsply Caulk, Milford, DE, USA) were

created to simulate overlying laboratory-processed composite resin restorations that had one surface airborne-particle abraded using 50-μm aluminum oxide particles (Asfer Industria Quimica Ltd., Sao Caetano do Sul, Brazil) for 10 s (distance from the tip: 1.5 cm; Bio-Art, São Carlos, Brazil). The dentin surfaces were acid etched with 35% phosphoric acid (Brazil Dentsply, Rio de Janeiro, Brazil) for 15 s and thoroughly water-rinsed. The excess water was removed using an absorbent paper. Two dual-polymerizing cementing systems (bonding agents / LAs) were used (Table 1):

- Excite DSC/Variolink II (Ivoclar Vivadent, Schann, Liechtenstein) and
- XP Bond/self-cure activator/Calibra (Dentsply Caulk, Milford, USA).

The adhesive systems were applied and light activated (Optilux 501; power density: 650 mW/cm^2, Demetron Kerr, Danbury, CT, USA) according to the manufacturers' instructions. Curing light intensity was continuously measured using a radiometer (Cure Rite, Dentsply Caulk). The LAs were applied at 25°C or were heated to 50°C prior to their use and were exposed to light from the same light-curing unit used to polymerize the bonding agents (dual-curing mode), or were allowed to self-cure (self-curing mode), resulting in 8 experimental groups (n = 5). For experimental groups using LAs heated to 50°C, base and catalyst pastes were equally dispensed on a glass plate laying on a heating stirrer surface (Cientec, Piracicaba, Brazil) that was previously set to 50°C. Cement and glass plate temperatures were continuously measured using a K-type thermocouple (Novus, Porto Alegre, Brazil) to ensure that both pastes reached 50°C. Based on the temperature control as measured using the thermocouple, it took approximately 30 s to 40 s for the LAs to reach 50°C. Only a small portion of the LA to be used for the cementing procedure was heated immediately before use; therefore, only one LA was heated at a time.

The resin composite discs were also placed on the heated plate for approximately 1 min; therefore, both LA and indirect restoration had the same temperature during cementation. The LAs were

Table 1 - Brand, composition, and batch number of the dual-cured cementing systems.

Product (Manufacturer)	Composition (manufacturer supplied) (Batch number)
Calibra / XP Bond (Dentsply Caulk)	**Base paste:** barium boron fluoroalumino silicate glass, Bis-GMA resin, polymerizable dimethacrylate resin, hydrophobic amorphous fumed silica, titanium dioxide, other colorants are inorganic iron oxides **Catalyst paste:** barium boron, fluoroalumino silicate glass, Bis-GMA resin, polymerizable dimethacrylate resin, hydrophobic amorphous fumed silica, titanium dioxide, benzoyl peroxide. (Base: 081105; Catalyst: 0812011) **Bonding agent:** HEMA, tert-butyl alcohol, dipentaerythritol pentaacrylate phosphate, acrylates, carboxylic acid modified dimethacrylate, urethane dimethacrylate resin, triethyleneglycol dimethacrylate (Lot: 0804002271) **Self-cure activator:** aromatic sodium sulfinate (self-cure initiator), acetone, ethanol (Lot: 041110)
Variolink II / Excite DSC (Ivoclar - Vivadent)	Paste of dimethacrylates, inorganic fillers, ytterbiumtrifluoride, initiators, stabilizers and pigments, Bis-GMA, TEGDMA, urethane dimethacrylate, benzoyl peroxide (Base: L46354; Catalyst: L 36656) **Bonding agent:** mixture of dimethacrylates, alcohol, phosphonic acid acrylate, HEMA, SiO_2, initiators and stabilizers (Lot: 59594)

TEGDMA: triethylene glycol dimethacrylate; Bis-GMA: bisphenol A diglycidyl ether methacrylate; HEMA: hydroxyethyl methacrylate; SiO_2: silicon dioxide.

mixed on the heated glass plate and were applied to abraded resin disc surfaces. For the room temperature groups (25°C), the same procedures were performed without heat. For the dual-cured groups, the disc was positioned and fixed to the adhesive-coated dentin surfaces under a 500-g load for 5 s, and a light-polymerizing unit tip was placed against it. The LA layer was then exposed to a curing light for 40 s. For the self-cured groups, the disc laying on the adhesive-coated dentin surface was subjected to the same load for 5 min. A 3-mm layer of self-curing composite resin (Alpha Plast, DFL Industria e Comercio S.A., Rio de Janeiro, Brazil) was placed on an untreated, pre-cured resin disc surface to facilitate specimen gripping during the testing process.

Microtensile bond strength test (μTBS)

The restored teeth were dark-stored in relative humidity at 37°C for 7 days. The teeth were sectioned vertically and serially into several 1.0-mm slabs using a diamond saw (Buehler Ltd, Lake Bluff, USA). Each slab was further sectioned to produce beams with a cross-sectional area of approximately 1.0 mm² at the bonded interface. Each bonded stick was attached to the testing jig grips with cyanoacrylate glue (Loctite Super Bonder Gel; Henkel, Düsseldorf, Germany) for μTBS testing using a universal testing machine (EZ Test; Shimadzu Co., Kyoto, Japan) at a crosshead speed of 1 mm/minute until failure. After testing, the specimens were carefully removed from the fixtures with a scalpel blade, and the cross-sectional area at the site of the fracture was measured to the nearest 0.01 mm using a digital micrometer (Mitutoyo America Corp., Aurora, IL, USA). Specimen cross-sectional area was calculated to produce μTBS data in units of stress (MPa). Five beams were tested per tooth, and μTBS average obtained from these beams represented the value for each restored tooth. Therefore, the experimental unit was the restored tooth.

Statistical analysis

Two-way analysis of variance (ANOVA) (temperature and polymerizing mode factors) was performed for each product. The statistical tests indicating significant differences were followed by Tukey HSD post hoc test at a preset alpha of 5%. The statistical analyses were performed using software (SAS for Windows V8; SAS Institute, Cary, NC, USA). Post hoc power analysis was performed for the statistical analysis of the μTBS data using statistical software (IBM SPSS 19, SPSS Inc., IBM Company, Armonk, NY, USA).

Failure mode analysis

All fractured surfaces of the tested specimens were allowed to air-dry overnight at 37°C, sputter coated (MED 010-Balzers, Balzers, Liechtenstein) with a thin gold layer, and examined using a scanning electron microscope (SEM - LEO 435 VP; LEO Electron Microscopy Ltd, Cambridge, UK). The failure patterns were classified as follows:

- adhesive along the dentin surface,
- cohesive within the dentin,
- cohesive within the LA and
- mixed, when simultaneously exhibiting the remnants of both the hybrid layer and the LA.

Results

Microtensile bond strength

Post hoc power analysis demonstrated statistical power greater than 95% at a pre-set alpha of 0.05. For Calibra, no significant differences in any factors were detected by ANOVA. In contrast, for Variolink II, ANOVA detected significant differences for the temperature ($p = 0.05$) and activation mode ($p < 0.0001$) factors. No significant difference in µTBS values was noted between teeth that were restored using Calibra at 50°C and 25°C, regardless of the activation mode (Table 2). Additionally, for all temperatures, no significant µTBS difference was observed between the dual- and the self-curing groups. In contrast, Variolink II at 50°C promoted

higher µTBS values than at 25°C in both activation modes ($p = 0.05$), whereas the dual-curing mode promoted higher µTBS values than the self-curing mode regardless of temperature ($p < 0.0001$).
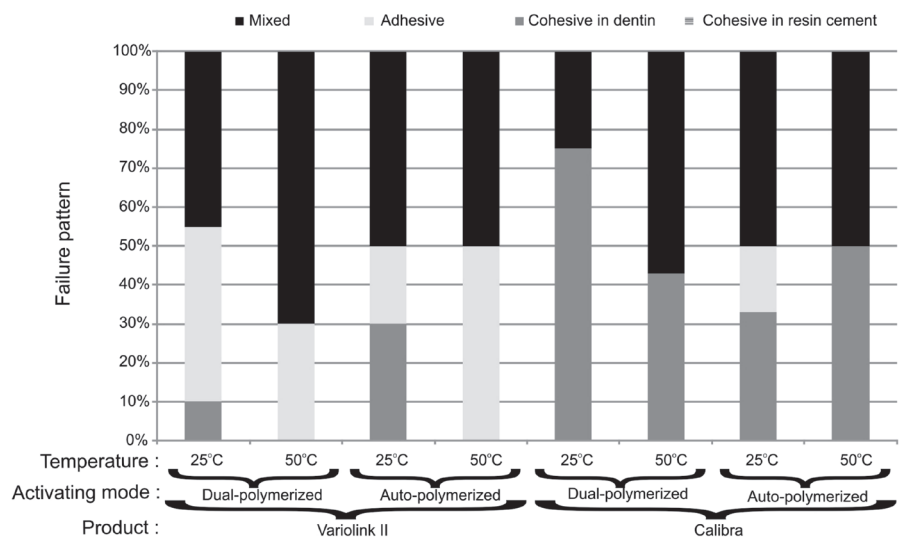
Failure pattern analysis

The distribution of failure modes among groups is shown in Figure 1. Figures 2A and 2B show SEM images of a mixed failure that was predominantly observed in the experimental groups. Only dual-polymerizing Calibra groups polymerized at 25°C exhibited failures predominantly located within the dentin. A decrease in cohesive failures within dentin was observed when Calibra was heated to 50°C. For Variolink II, the cohesive failures within dentin in both the dual-polymerizing and auto-polymerized groups at 25°C were eliminated when both groups were polymerized at 50°C.

Table 2 - µTBS values of dual-cured LAs submitted to different curing modes and temperatures.

		Dual-polymerized	Self-polymerized
Variolink II	25°C	33.38 (8.74) Ba	17.34 (9.07) Bb
	50°C	47.12 (8.58) Aa	21.65 (10.20) Ab
Calibra	25°C	37.45 (9.64) Aa	28.58 (8.94) Aa
	50°C	36.17 (8.07) Aa	28.83 (10.10) Aa

Different letters (upper case letter within column; lower case letter within row) indicate significant difference among the means. No comparison was made between products.

Figure 1 - Failure pattern of fractured specimens from all experimental groups.
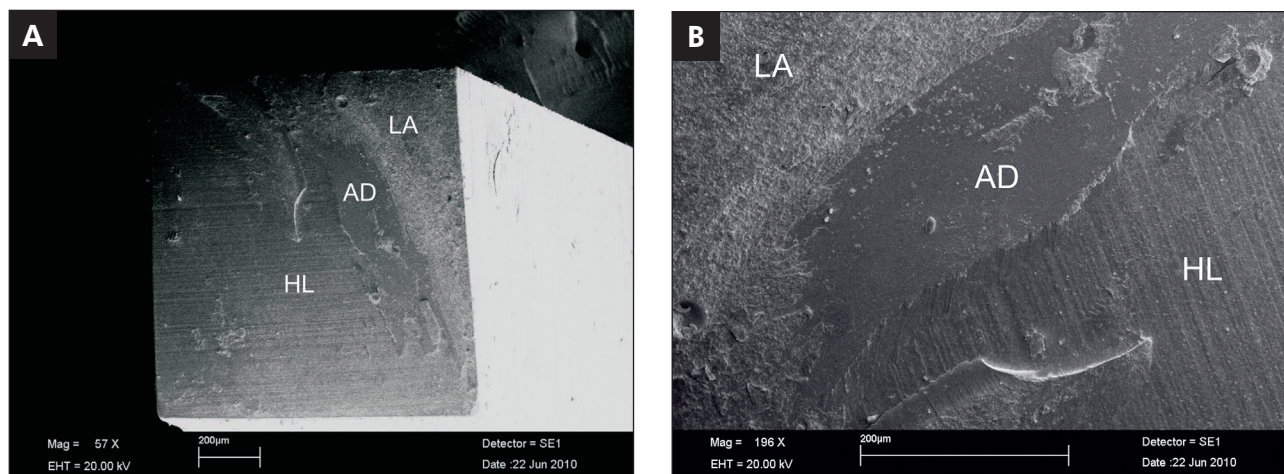
Figure 2 - Illustrative scanning electron microscope image of a mixed failure pattern at a lower (**A:** 57×) and a higher (**B:** 196×) magnification to exhibit the hybrid layer (**HL**), the adhesive layer (**AD**), and the luting agent (**LA**).

Discussion

In this study, the temperature of 50°C was chosen based on previously reported data.[12,13,18] However, it must be emphasized that a temperature decline is expected, similar to the temperature decline observed in the preheated composite resins that are placed in tooth cavities.[16] For this reason, the indirect restoration was also heated to delay any reduction in the LA temperature. Therefore, the present results should not be extrapolated to clinical situations in which the LA is heated and the indirect restoration is maintained at room temperature. Moreover, any comparison between products was neglected because the evaluated resin cements use different bonding agents. For this reason, if product comparisons were made, the possible differences in the µTBS values could be related to the differences in bonding agent compositions rather than to increased LA temperature.

Variolink II at 50°C promoted higher µTBS than at 25°C in both curing modes. Therefore, the research hypothesis was accepted for this product regardless of the activation mode. This finding is in agreement with those of studies that evaluated preheated LAs,[22,23] and demonstrates that the previously shown[24] increased LA monomer conversion and improved mechanical properties do increase bond strength to dentin. Possibly, decreased resin viscosity and high radical mobility were not the only factors contributing to the greater bond strength.

The self-curing component benzoyl peroxide (BPO) from Variolink II is unstable and can be activated by heat.[25,26] Thus, BPO decomposes faster at higher temperatures and creates more radicals more rapidly than it does at room temperature.[26] As a consequence, more radical formation may have contributed to the increased µTBS values. Moreover, the use of this LA at a high temperature also reduced the percentage of the cohesive failures within the dentin. Therefore, a high temperature could have influenced the mechanical properties at the bonding interface below the LA layer. However, further studies evaluating such effects are needed to confirm this observation.

Regardless of temperature, lower µTBS was observed when Variolink II relied solely on the self-polymerizing components rather than when the LA was exposed to curing light. Therefore, the self-curing mode was less effective than the dual-curing mode, even at a high temperature. This finding might be attributed to the low BPO content in Variolink II (information from the manufacturer's material safety data sheet), which explains why this product is not indicated in clinical situations in which the activating light is entirely blocked by an indirect restoration.[10]

Conversely, Calibra was not influenced by heat because no significant difference in the µTBS values was noted between the groups polymerized at 25°C and 50°C. Therefore, the research hypothesis was

rejected for this LA. Curiously, at 25°C, no significant difference in the μTBS was noted between the dual- and self-curing groups, indicating that the self-curing mode in Calibra was as effective as the dual-curing mode. This finding might be attributed to the higher BPO content (approximately 2%) in Calibra compared to Variolink II (approximately 1%), according to the manufacturers' material safety data sheets. Because an increased temperature decomposes BPO more quickly, LAs with higher BPO content at a high temperature may set faster than those with low BPO content. This was observed during specimen preparation when Calibra started setting immediately after the indirect restoration was cemented onto the tooth. Therefore, the polymer network may have been rigid during the light exposure. Thus, any possible benefit from a high temperature on the bond strength was masked by the tension cre-ated at the bonding interfaces caused by the premature LA setting. This observation confirmed previous findings.[22,23] For this reason, the use of Calibra at 50°C is concerning because its working time was apparently not sufficient to allow for a proper seating of the indirect restoration on the prepared tooth. For this reason, manufacturers should provide more detailed information regarding the content of the self-curing components to allow clinicians to choose when to heat LAs prior to their application.

Conclusion

An increased temperature prior to polymerization can promote higher μTBS to dentin in indirect restorations. However, the effectiveness of LA preheating on μTBS was product-dependent because these effects were observed in the LAs with a low content of self-curing components.

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