Effects of cement-curing mode and light-curing unit on the bond durability of ceramic cemented to dentin

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Abstract: The aim of this study was to evaluate the effects of different light-curing units and resin cement curing types on the bond durability of a feldspathic ceramic bonded to dentin. The crowns of 40 human molars were sectioned, exposing the dentin. Forty ceramic blocks of VITA VM7 were produced according to the manufacturer's recommendations. The ceramic surface was etched with 10% hydrofluoric acid/60s and silanized. The dentin was treated with 37% phosphoric acid/15s, and the adhesive was applied. The ceramic blocks were divided and cemented to dentin according to resin cement/RC curing type (dual- and photocured), light-curing unit (halogen light/QTH and LED), and storage conditions (dry and storage/150 days + 12,000 cycles/thermocycling). All blocks were stored in distilled water (37°C/24h) and sectioned (n = 10): G1 - QTH + RC Photo, G2 - QTH + RC Dual, G3 - LED + RC Photo, G4 - LED + RC Dual. Groups G5, G6, G7, and G8 were obtained exactly as G1 through G4, respectively, and then stored and thermocycled. Microtensile bond strength tests were performed (EMIC), and data were statistically analyzed by ANOVA and Tukey's test (5%). The bond strength values (MPa) were: G1 - 12.95 (6.40)[ab]; G2 - 12.02 (4.59)[ab]; G3 - 13.09 (5.62)[ab]; G4 - 15.96 (6.32)[a]; G5 - 6.22 (5.90)[c]; G6 - 9.48 (5.99)[bc]; G7 - 12.78 (11.30)[ab]; and G8 - 8.34 (5.98)[bc]. The same superscript letters indicate no significant differences. Different light-curing units affected the bond strength between ceramic cemented to dentin when the photocured cement was used, and only after aging (LED > QTH). There was no difference between the effects of dual- and photo-cured resin-luting agents on the microtensile bond strength of the cement used in this study.

Descriptors: Resin Cements; Ceramics; Dentin.

Introduction

In association with the development of ceramic materials, resin cements also demonstrate increased improvement in their chemical and mechanical characteristics. These cements can be divided according to the type of activation:
- self-,
- light-, or
- dual-curing.

The dual resin cement presents reagents of both forms of polymeriza-

tion.[1] The chemical component ensures adequate polymerization in the areas of the cement that do not receive sufficient light to initiate polymerization.[2] Many studies have reported that the resin cements with dual polymerization are extremely dependent on photoactivation, since chemical polymerization itself is insufficient to complete the polymerization of these cements.[3]

The degree of conversion (DC) depends on the energy supplied during composite polymerization, characterized as the product of luminous intensity and time of exposure.[4,5] Reduced microhardness can be synonymous with incomplete resin cement polymerization, causing a reduction in the mechanical properties of the cement, increasing water absorption[6] and favoring microinfiltration.[7] Moreover, unpolymerized molecules can be leached from the material, causing inflammation.[8] Therefore, it is important to optimize the photoactivation of resin cements to improve the clinical performance of these materials. Fan *et al.*[9] affirmed that the periods of light exposure recommended by resin manufacturers are not always adjusted to provide complete polymerization.

The clinical performance of feldspathic ceramic restorations is directly related to the cementing procedure, since it involves the union between different substrates (ceramic and dental structure).[10] For this adhesive process, the dental and ceramic surfaces must be adequately conditioned. Additionally, there must be adequate polymerization of the resin cement agent, since it is essential in preventing microleakage and allowing for greater longevity of the restoration.

Thus, the objective of the current study was to verify the effects of different light-curing units and different types of polymerization of the cement agent on the bond durability of a feldspathic ceramic cemented to dentin. The appropriate photoactivation of the resin cement is considered an essential factor for adequate bond strength at the ceramic-cement-dentin interface, as well as for optimization of the physical properties.[11] Our study hypotheses were that:

- (1) different light-curing units influence the bond strength of ceramic cemented to dentin,
- (2) dual-cure resin cement improves bond strength, and
- (3) storage affects bond strength values.

Methodology

This study was approved by the Research Ethics Committee of São Paulo State University, São José dos Campos, Brazil.

Forty non-carious human molars were extracted for periodontal or orthodontic reasons, and stored in distilled water. The crowns were transversely sectioned with a diamond saw at low speed and with intense water-cooling, exposing the mid-coronal dentin. The distance between the cemento-enamel junction and the flat dentin surface was kept to a similar depth for all specimens. Dentin surfaces were visually inspected for the absence of enamel. We created a standardized smear layer by wet-grinding the dentin surface flat with 600-grit silicon carbide paper for 60 s (Labpol 8-12, Extec, Enfield, USA).

Forty ceramic blocks (6.4 × 6.4 × 2.5 mm) of VITA VM7 (Base Dentin 2M3, Vita Zahnfabrik, Bad Säckingen, Germany, #31570, liquid 17501) were produced. The cementation surface of each ceramic block was leveled and polished with silicon carbide papers in sequence (600-, 800-, and 1200-grit) under water-cooling.

All ceramic blocks were ultrasonically cleaned for 5 min in distilled water. The cementation surfaces were etched with 10% hydrofluoric acid gel (Dentsply, Petrópolis, Brazil, #L806765) for 60 s, rinsed with water, and air-dried. The ceramics were ultrasonically cleaned in distilled water for 5 min. Then, the silane was applied (Monobond-S, Ivoclar Vivadent, Schaan, Liechtenstein, #H24764) and allowed to sit for 5 min.

The coronal dentin was treated with a multi-step total-etch adhesive system (Excite DSC, Ivoclar Vivadent, Schaan, Liechtenstein, #H23024). The dentin surface was air-dried and etched with 37% phosphoric acid (Dentsply, Petrópolis, Brazil, #040707) for 15 s and washed, and then the excess water was removed with absorbent paper. Two layers of the adhesive system were applied to the coronal dentin. The specimens were gently air-dried and light-cured for 20 s, by means of a QTH (XL 3000, 3M/ESPE, St. Paul, USA;

light intensity: 600 mW/cm²) or a LED (SmartLite PS/ Dentsply, Petrópolis, Brazil, light intensity: 950 mW/cm²).

The 40 ceramic blocks were randomly divided into four groups (n = 10) based on the factors "light-curing units" (QTH and LED) and "resin cement" (Variolink II photo- and dual-cured, Ivoclar Vivadent, Schaan, Liechtenstein; Base, G24884; Catalyst, J09824). The cement was placed on the treated ceramic and coronal dentin surfaces, following the manufacturer's instructions. Each ceramic block was placed on the dentin surface under a load of 750 g for 5 min. The resin cement was light-activated by the light-curing units QTH or LED for 40 s, according to the experimental group assignment. The specimens from all groups were stored in distilled water for 24 h at 37°C prior to microtensile bond strength (MTBS) testing.

The specimens were sectioned by means of a diamond disc, under water cooling (LabCut 1010, Extec, Enfield, USA). Approximately 10 specimens were obtained from each block. The beam specimens had non-machined (non-trimmed) bonding areas measuring approximately 1.0 ± 0.1 mm² and a length of 6 mm. The specimens were randomly divided into 2 testing conditions. In the dry condition (Dry), specimens were immediately subjected to MTBS after being sectioned, while in the aged condition, specimens were subjected to thermocycling (TC: 12,000 cycles; 5°C–55°C) (Nova Etica, São Paulo, Brazil) and storage (150 days) in distilled water at 37°C, and then tested.

Therefore, 8 groups were obtained (n = 10):
- G1 - QTH + dual-cured resin cement + Dry;
- G2 - QTH + photo-cured resin cement + Dry;
- G3 - LED + dual-cured resin cement + Dry;
- G4 - LED + photo-cured resin cement + Dry;
- G5 - QTH + dual-cured resin cement + TC;
- G6 - QTH + photo-cured resin cement + TC;
- G7 - LED + dual-cured resin cement + TC; and
- G8 - LED + photo-cured resin cement + TC.

The MTBS was performed in a universal testing machine (EMIC DL-1000, EMIC, São José dos Pinhais, Brazil). The specimens were analyzed under an optical microscope (Mitutoyo, Measuring Microscope MFA - Series 5051H, Suzhavo, Japan). The failure types were classified according to the following scores:[12]
- Score A, adhesive failure along the interfacial region between the dentin and resin cement;
- Score B, cohesive failure in the dentin;
- Score C, cohesive failure in the resin cement; and
- Score D, mixture of adhesive failure and cohesive fracture of the dentin or resin cement.

In addition, some representative specimens from each group were analyzed under SEM (LEO-1450VP/LEO-Zeiss, Oberkochen, Germany).

The MTBS data were analyzed by three-way analysis of variance (ANOVA) and Tukey's multiple-comparison tests (5%). P-values less than 0.05 were considered to be statistically significant in all tests.

Results

Three-way ANOVA revealed that the MTBS was significantly affected by light-curing units (p = 0.0037 < 0.05) and storage conditions (p = 0.0001 < 0.05). The interaction among the light-curing unit, resin cement, and storage factors was statistically significant (p = 0.0030).

Table 1 and Figure 1 present the MTBS results for the experimental groups. When the storage was

Table 1 - Mean (± SD) bond strength values (MPa) for light-curing units, resin cement, and storage combinations [*The same superscript letters indicate no significant differences (Tukey's test, $\alpha = 0.05$).]

Experimental groups	Mean (SD)
Group 1	12.95 ± 6.40[ab]
Group 2	12.02 ± 4.59[ab]
Group 3	13.09 ± 5.62[ab]
Group 4	15.96 ± 6.32[a]
Group 5	6.22 ± 5.90[c]
Group 6	9.48 ± 5.99[bc]
Group 7	12.78 ± 11.30[ab]
Group 8	8.34 ± 5.98[bc]
Mean (SD)	4.4 ± 1.9

* Gr1 to Gr4 = without thermocycling; Gr5 to Gr8 = with thermocycling.

Figure 1 - Interaction of bond strength mean values (MPa) according to light-curing unit, resin cement, and storage conditions.
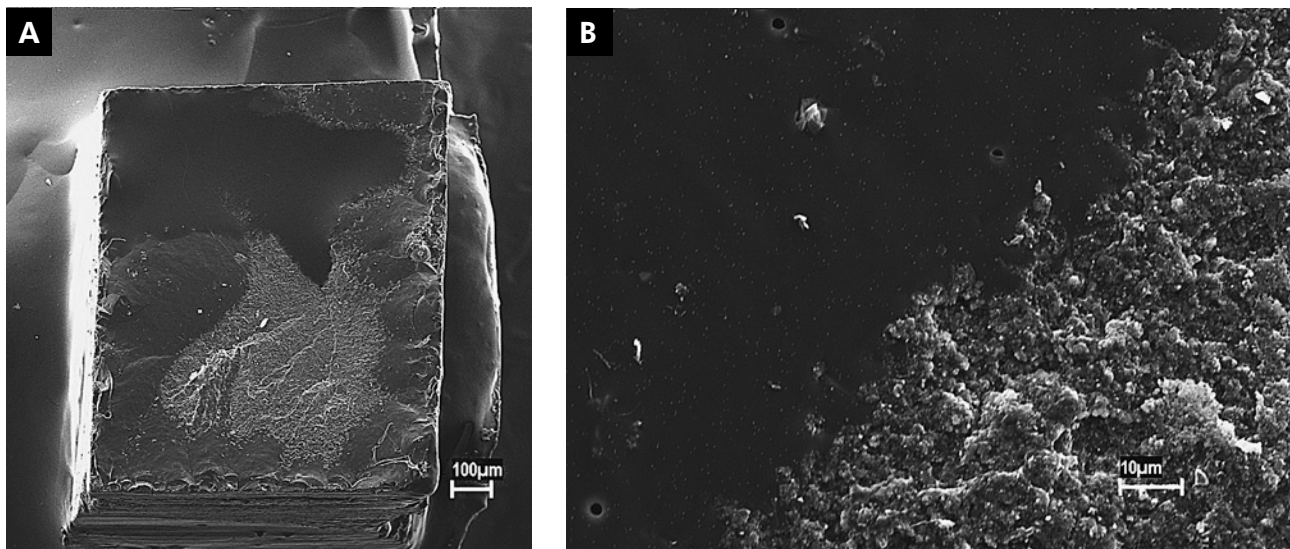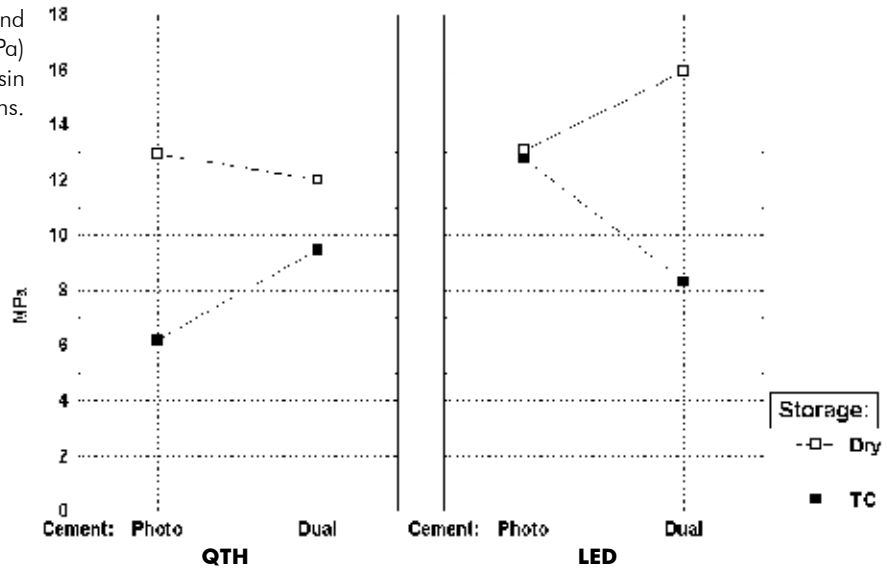

Figure 2 - Representative micrographs of a mixture failure of a specimen from G4 (**A**, 64× and **B**, 1000×) after MTBS testing.

analyzed, the dry condition presented values significantly higher when compared with the thermocycling condition in groups G1/G5 and G4/G8. Similar results were found when the light-curing units were compared, except for G5/G7. In contrast, when the resin cement was analyzed, the values of photo- and dual-cured cement were similar.

Regarding fracture analysis, most specimens presented mixed failures. No scores of A and C were observed. A representative micrograph of a mixed fracture is presented in Figures 2A and 2B.

Discussion

Different factors, including the type and chemical composition of the resin matrix (filler type or filler load), determine the mechanical properties of resin-luting agents,[11] and also lead to variations in bond strength to dentin. Therefore, only one dual-polymerized resin-luting agent was chosen for the present study, and it was used with a catalyst (dual polymerization mode) to eliminate the previously mentioned material-dependent properties. Variation in tubule density at different dentin depths

is another significant factor that influences bond strength.[13]

The first and third hypotheses were partially accepted:

- (1) different light-curing units influence the bond strength of the ceramic cemented to dentin only for the photo-cured resin cement after aging, and
- (3) storage did not affect the bond strength values of all groups.

The second hypothesis was not validated: There was no difference between the effects of photo- and dual-cured resin cement on bond strength.

When one considers the direct relationship between polymerization of the resin-luting agent and its bond strength to dentin, the risk of debonding is higher immediately following cementation, because the cement has not yet reached the highest degree of polymerization. If some factor prevents the light from reaching the cement layer, thereby causing insufficient polymerization, then areas of cohesive failure in the resin-luting agent should be detected in SEM analysis. In the present study, the SEM observations revealed mixed failures (Figures 2A, 2B). Hofmann *et al.*[14] verified that flexural strength, modulus, and hardness of self-cured Variolink II reached 68.9%, 59.2%, and 91.1%, respectively, of the corresponding values obtained by dual-curing with direct irradiation.

Adequate curing of resin-luting materials may also be a problem under ceramic restorations, especially when these resins are used without a catalyst.[14,15] In previous studies, the use of a self-curing catalyst resulted in a higher degree of polymerization when compared with that achieved by light polymerization alone.[7] However, in the present study, no difference was found between the resin cement curing modes evaluated in terms of bond strength. El-Mowafy and Rubo[16] showed that dual-cured resin cements reached an adequate DC, even in areas that are less accessible to the curing light, emphasizing the importance of the self-curing catalyst. In contrast, chemical curing alone of Variolink II produced lower values of surface hardness when compared with those achieved by light- or dual-curing.[11,16] Photo-curing

of dual-cured resin-luting agents provides an initial fixation for restorations, with chemical-curing responsible for the final bonding.[17] In contrast, the results of the present study showed that the use of a catalyst of the resin cement studied produced no difference on the bond strength when compared with the bond strengths obtained with light-curing.

Another consideration in relation to the degree of polymerization of resin-luting agents is the intensity of the light-curing unit. In several studies, the curing efficacy of halogen and LED curing units has been compared.[7,14,18-24] LED curing units provide efficient energy, with a peak length of about 470 nm required by the photo-initiator, camphorquinone.[25] It has generally been found that light intensity, not the light-curing unit itself, was the decisive factor determining the DC and resulting in the expected physical and mechanical properties of resin composites.[5] The ISO-recommended intensity for polymerization lights is 300 mW/cm², and the standard requirement for depth of polymerization is 1.5 mm.[9] Light intensities of 600 mW/cm² and 950 mW/cm² were used for polymerization in the current study. Polymerization of light-polymerized resin-luting agents for all-ceramic restorations may be incomplete when low-output polymerization units are used.[15] For this reason, a high-intensity light-curing unit and longer polymerization times may directly affect bond strength values. Feng *et al.*[26] suggested at least doubling the photocuring time recommended by the manufacturers.

The light-curing units under investigation presented similar results except for the photo-curing resin cement groups after aging. It can be initially assumed that differences between the activation modes occurred because of differences in the mechanical strength of the resin cements. In fact, previous studies have shown that Variolink II presents a lower DC and hardness in the self-cure mode when compared with the dual-cure mode.[7,11] In other studies,[2,16] it was reported that Variolink II has a relatively weak chemical polymerizing component and relies mainly on its light-polymerizing capability.

The present study examined the early strength and durability of the Vita VM7 ceramic sample bonded to dentin. An important factor related to water sorption of resin-based composite materials would appear to be the degree of polymerization. Water sorption itself may not cause significant problems, but if it is associated with incomplete polymerization, the long-term success of a restoration may be compromised. The results of the present study demonstrated that thermocycling (12,000 cycles) and 150 days of storage did not result in decreased MTBS values in all groups. These findings are supported by those of other studies.[27] Another study[21] indicated that the type of surface-conditioning agent, rather than the type of resin-luting agent, predominantly influenced the bond strength to ceramic.

Numerous factors—such as optical properties of the ceramic material,[28] the types and chemical compositions of the resin-luting agents,[29] and the use of different cements[30] and ceramic shades—may influence the polymerization degree of resin-luting agents. Therefore, it was not possible to include all of these variables in the same *in vitro* study. However, according to the results of this study, LED and QTH units may be used to light-cure resin cement when bonding feldspathic ceramic restorations to dentin.

Conclusions

Based on our results, we concluded that different light-curing units influence the bond strength of ceramic cemented to dentin when photo-cured cement is used, and only after aging (LED > QTH). In addition, there was no difference between the effects of dual- and photo-cured resin-luting agents on the MTBS of the cement used in this study. Thermocycling affected bond strength values.

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