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ORIGINAL ARTICLE

Relationship between bond strength and marginal and internal adaptation of composite restorations photocured by different methods

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Abstract

Objective. This study evaluated the relationship between bond strength and marginal and internal adaptation of composite restorations photocured using different methods with a quartz-tungsten-halogen light. ***Material and Methods.*** A push-out test was performed to evaluate bond strength of conical restorations in 50 bovine incisors. To evaluate marginal (external) and internal restoration adaptation, 50 circular all-enamel margin preparations were done in bovine incisors. For both tests, the preparations were filled with Esthet•X resin composite. Specimens were distributed into 5 groups ($n = 10$) depending on photoactivation method: G1: continuous light 700; G2: continuous light 150; G3: soft-start; G4: intermittent light; and G5: pulse-delay. The energy density for each method was standardized: 14 J/cm^2. Caries Detector® (Kuraray) was placed in restoration margins for detection of marginal adaptation. The percentage of interfaces present as gaps was determined using digital images. Specimens were then sectioned, stained, and the internal adaptation was recorded in a similar manner. Data were submitted to ANOVA and the Tukey HSD test, pre-set $\alpha = 0.05$. ***Results.*** Bond strength G5 (7.2 MPa $\pm$ 1.3) was significantly greater ($p = 0.00280$) than G1 (4.6 MPa $\pm$ 1.5). G2, G3, and G4 showed equivalent, intermediate strength values. No significant difference was found in marginal adaptation of any of the groups ($p = 0.16911$). Internal adaptation results were the inverse of strength results: G5 (2.8% $\pm$ 4.9) showed significantly less ($p = 0.00979$) gap formation compared to G1 (10.1% $\pm$ 6.2). ***Conclusion.*** Some modulated photocuring methods can increase bond strength while decreasing internal gap formation. An inverse relationship was found between push-out bond strength and internal adaptation. Marginal adaptation was not affected by any photoactivation method.

Key Words: *Bond strength, composites, gap formation, stepped polymerization*

Introduction

Although light-cured resin composites have become the material of choice in directly restoring anterior and posterior teeth, these materials undergo significant volumetric shrinkage when polymerized [1]. *In vitro* measurements of composite polymerization range from 1.9% to 6% [2]. Placement and bonding of composites in preparations induces development of mechanical stress inside the material as well as at the bonded interface [3]. Stress is also transmitted via bonded interfaces to tooth structures [3]. The rapid conversion rate in light-cured composites quickly induces an increase in composite stiffness, causing high shrinkage stresses at the bonded interface [4]. This stress may disrupt bonding between the composite and the preparation walls, or even cause cohesive failure of either the restorative material or the surrounding tooth tissue [3,4]. This stress development is the main cause of marginal failure and subsequent leakage in resin composite restorations [5].

Compensatory curing methods to counteract stress development have been proposed. Minimizing stress may potentially reduce gap formation [1,5–7]. These alternative photoactivation methods (modulated polymerization) have been shown to have beneficial effects [7–12]. A reduction in curing rate results in slower development of composite stiffness, and allows the material to flow instead of becoming rigid, thus reducing shrinkage stress [9,11,13]. Previous studies [1,14] have shown that marginal adaptation of composites can be improved by

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light-curing with a low power density. Conversely, high light intensity is necessary to achieve deep and maximal polymerization of the material [15]. Therefore, modulated photoactivation methods, such as soft-start, intermittent light, and pulse-delay, have been proposed [7–12].

Resin composites photocured using the soft-start technique demonstrate substantially lower viscosity increase, allowing more material flow during the earlier stages of curing, while keeping the total exposure time reasonably short [7,10]. Soft-start photoactivation has been shown to provide better cavo-surface marginal adaptation of composite restorations [7]. This photoactivation method affects shrinkage, surface hardness, and residual monomer concentration similarly as in conventional, continuous higher intensity photoactivation, so long as the total energy dose is the same [8,13].

The intermittent light photoactivation method consists of cyclic application of light-on and light-off periods (a continuous pulse). This method has been shown to effectively reduce polymerization shrinkage. The light-off period may modify polymerization kinetics by reducing or modifying the distribution of stress [16].

The pulse-delay photoactivation method is a combination of low energy density application followed by a lag period (delay) before a final high power density exposure is provided [17,18]. The initial, low energy, density provides enough light to allow the polymerization reaction to start. During the delay period, the polymerization process is slow, because no new radicals are forming, and the reaction progresses as a result of the "dark cure" reaction [19]. At the last stage, a final light exposure to high power density ensures maximal composite cure, thus providing similar physical and mechanical properties to the restoration as if cured using a continuous light exposure method [17].

The ultimate goal of a composite restoration is to ensure the biomechanical integrity of the tooth and this implies achieving and maintaining proper function over time. The same is derived from satisfactory marginal and internal restoration/tooth adaptation. The presence of gaps is considered the first sign of restoration failure, and can be demonstrated clinically by marginal staining. Thus, identification of early marginal changes can determine the prognosis for longevity of composite restorations.

The presence of internal gaps between the material and tooth structure causes fluid flow in the dentin tubules, resulting in typical postoperative sensitivity. It is important to enhance the adhesion between dentin and resin composite because improved bond strength not only prevents postoperative sensitivity, but also leads to better restoration retention.

The relationship between bond strength and gap formation is controversial in the literature [20]. Intuitively, it seems that when bond strength is increased the ability of the material to withstand the forces that might stress the interface and disrupt the adaptation of restorative material to the cavity walls is enhanced. However, several studies [6,21–23] have shown the absence of any relationship between higher bond strength and improved marginal seal or decreased microleakage levels. The lack of any relationship between improved bond strength and reduced gap formation is explained on the basis of bond maturation [20]. In most bond strength tests, the preparations used have a C-factor of about 0.2, compared to 3 to 5 in typical Class V, I, or II cavity preparations [24,25]. Therefore, the increased polymerization shrinkage stress encountered in cavity preparations could cause bond disruption that might not occur in the bond strength specimen. Also, polymerization shrinkage forces will occur very rapidly and stress the bond before it has had the chance to mature [26], causing bond disruption that will result in early gap formation [20].

The purpose of this research was to evaluate the influence of alternative (modulated) photoactivation techniques (continuous low power, soft-start, intermittent light, and pulse-delay) on marginal and internal adaptation of resin composite restorations and on bond strength of composite restorations using the push-out test when each treatment, as well as the control, received the same total energy density dose. In the control group, the specimens were subjected to conventional intensity and continuous light. It was hypothesized that the alternative methods would significantly reduce marginal and internal gap formation and increase bond strength values relative to the control. It was further hypothesized that there would be an inverse relationship between push-out bond strength values and marginal and internal gap formation.

Material and methods

Marginal and internal adaptation

Fifty bovine incisors were selected and cleaned immediately after extraction. They were stored in a 0.5% aqueous solution of Chloramine T (Mallinckrodt Baker Brazil, 04795-100, São Paulo, Brazil) at 4°C for no more than a week to avoid bacterial growing. After removing the root portion 1 mm below the cemento-enamel junction, the buccal tooth surface was ground to provide at least a 6 mm diameter flat area using a water-cooled mechanical polisher (Minimet 1000; Buehler Co., UK and Lake Bluff, Ill., USA) using 320-, 400- and 600-grit silicone carbide (SiC) abrasive paper (Carbimet Disc Set, #305178180; Buehler). The teeth were examined under a stereomicroscope (Zeiss, Manaus, AM, Brazil) at 25 × to verify that only enamel was present on the surface.

Preparations (4 mm diameter × 1.5 mm deep) were prepared in the central area of the flattened surfaces using a round tip diamond bur (# 3053; K. G. Sorensen, São Paulo, SP, Brazil) mounted in a high-speed handpiece (Kavo, Joinville, SC, Brazil), under constant air-water cooling. The burs were replaced after every 10th preparation.

Internal preparation walls were 90° to the enamel surface plane, while the internal line angles were rounded using the diamond end. The C-factor of the preparation was calculated to be 2.5. The specimen was discarded if any pulp exposure was noted at the axial wall during preparation.

An adhesive system (Single Bond, lot # 4KF; 3M/ESPE Dental Products, St. Paul, Minn., USA) was applied in accordance with the manufacturer's instructions. Preparation walls were etched with 35% phosphoric acid (H_3PO_4) gel (lot # 4CB; 3M/ESPE) for 15 s, rinsed for 10 s, and blotted dry using absorbent paper. The adhesive system was applied twice with a 5-s interval in between, dried carefully for 15 s with air to remove solvent, and light-cured for 10 s using a quartz-tungsten-halogen light-curing unit (XL 2500; 3M/ESPE). The power density of the light was measured using a hand-held curing radiometer model 100 (Demetron Research Corp., Danbury, Ct., USA) and found to be 700 mW/cm^2. Power density readings were performed throughout the experiment to ensure proper light performance. A commercial hybrid composite (Esthet-X, shade A3, lot # 0308112; Dentsply De Trey, Konstanz, Germany) was inserted in a single increment, and a Mylar strip was placed over the composite. A microscope slide was used to force the composite into the preparation and to extrude the excess material. The slide was then removed and the light-curing tip placed against the Mylar strip. The teeth were randomly assigned into five groups ($n = 10$) according to the photoactivation method, and exposure duration was adjusted to provide equivalent energy density dosages among treatments: 14 J/cm^2 (Table I).

The intermittent light photoactivation method used an experimental curing unit developed in the Dental Materials Department of the Piracicaba Dental School, UNICAMP. The unit was assembled from a commercial curing unit (Optilux 150 – Demetron) with a halogen light. This unit was modified electronically to provide cyclic, pulsed irradiation (2 s light-on and 2 s light-off) [9,16].

The other photoactivation methods were conducted using the curing unit XL2500 (3M/ESPE). The power density of 150 mW/cm^2 used in the soft-start and pulse-delay photoactivation methods was obtained using a standard separator (black acrylic cylinder 1.2 cm in height and 1.0 cm in diameter).

After light-curing, specimens were stored in distilled water at 37°C for 24 h and then finished and polished under running water using 600- and 1200-grid SiC sandpaper.

In order to determine surface marginal adaptation, a 1.0% acid red propylene glycol solution (Caries Detector®; Kuraray Co., Osaka, Japan) was applied at the restoration margins for 5 s [7,9]. Specimens were then rinsed in tap water and gently blown dry. This technique stained any gaps, such that they could easily be quantified. Margins were evaluated using a stereomicroscope (MZ6; Leica Microsystems Ltd., Heerbrugg, Switzerland) at 16 ×. A digital image of each specimen was obtained. The accumulated length of dye-stained gaps along the restoration margins was measured (μm) from the images using software developed by the Department of Dental Diagnostic Science at the University of Texas Health Science Center (San Antonio, Tx., USA) (v. 2.0 – September 1997). The length of the gap formed was calculated as a percentage of the entire margin length.

After the evaluation of surface marginal adaptation, the restorations were cut into 1-mm-thick sections in a bucco-lingual direction using a water-cooled saw (ISOMET 1000; Buehler), to obtain 2 slices of each restoration. The Caries Detector® solution was applied to the internal restoration margins to identify interfacial gaps in a manner similar to that described above for marginal adaptation.

Marginal adaptation data were transformed (arc sen $\sqrt{x/100}$) due to the abnormal distribution of the data and submitted to ANOVA (pre-determined

Table I. Description of the photoactivation methods

Photoactivation method*	Light curing unit	Power density and exposure duration
Continuous light 700	XL 2500, 3M-ESPE	700 mW/cm^2 for 20 s
Continuous light 150	XL 2500, 3M-ESPE†	150 mW/cm^2 for 94 s
Soft-start	XL 2500, 3M-ESPE†	150 mW/cm^2 for 10 s + 700 mW/cm^2 for 18 s
Pulse-delay	XL 2500, 3M-ESPE†	150 mW/cm^2 for 5 s + 3 min with + 700 mW/cm^2 for 19 s
Intermittent light	Optilux 150, Demetron, adapted‡	600 mW/cm^2 (2 s light on + 2 s light off) for 56 s

*The energy density dose applied for all groups was 14 J/cm^2.

†Reduction of the power density in these groups was obtained using a standard separator.With this photoactivation method, an experimental curing unit developed in the Dental Materials Department, Piracicaba Dental School, UNICAMP was used. The unit was assembled from a commercial curing unit (Optilux 150 – Demetron) with a halogen light. This unit was modified electronically to provide cyclic, pulsed irradiation (2 s light on and 2s light off).

significance level of 5%). Internal adaptation data were submitted to ANOVA and the Tukey HDS post-hoc test at a pre-determined significance level of 5%.

Push-out bond strength

Fifty bovine incisors selected and cleaned immediately after extraction were stored in a 0.5% aqueous solution of Chloramine T at 4°C for no more than 1 week. The crowns were cut off at the cement–enamel junction using a double-faced diamond disk (K. G. Sorensen) (Figure 1A).

Conical preparations (top diameter of 5.0 mm, bottom diameter of 4.0 mm, and 2.0 mm in height; Figure 1C) were prepared in the buccal surface of each tooth using a diamond tipped bur (#3131; K. G. Sorensen) mounted in a high-speed handpiece (Kavo, Joinville, SC, Brazil) under constant air–water cooling in a standard cavity preparation appliance (Figure 1B). The diamond burs were replaced after every 10th preparation. The C-factor of the preparation was calculated to be 2.2.

A similar restorative procedure was performed as described above for the marginal and internal adaptation tests; an adhesive system (Single Bond, lot # 4KF; 3M/ESPE) used in accordance with the manufacturer's instructions. A commercial hybrid composite (Esthet•X, shade A3, lot # 0308112; Dentsply De Trey) was placed in a single increment (Figure 1C). The specimens were randomly assigned into five groups ($n = 10$) depending on photoactivation method and exposure duration (Table I).

After applying the light-curing procedures, specimens were stored in distilled water at 37°C for 24 h and then finished (Sof-Lex; 3M/ESPE) on the enamel surface to remove excessive material.

A diamond tipped bur (3017HL; Fava Metalúrgica, São Paulo, SP, Brazil) was used to selectively remove the lingual face of the crown, with the goal of exposing the bottom (axial) surface of the restoration. The mesial and distal crown segments on the lingual surface were preserved to reinforce the specimen (Figure 1D).

A push-out bond strength test was performed. An acrylic device with a central hole was attached to the base of a universal testing machine (Instron, model 4411; Buckinghamshire, UK). The central hole was used to control specimen positioning with the restoration bottom side up (smaller diameter of the preparation). A round stainless steel tip (2 mm diameter) attached to the upper, movable member of the testing machine (Figure 1E) applied a compressive force at a rate of 0.5 mm/min on the exposed, bottom (axial) surface of the restoration and forced the restoration out in a facial direction. The results were recorded in units of force and converted into units of stress (MPa) by dividing by the remaining, conical specimen bonded area.

After the test, the fractured specimens were examined under a stereomicroscope at 40 × (Carl Zeiss, Manaus, AM, Brazil) and classified in terms of failure: cohesive failure in composite, cohesive failure in dentin, adhesive failure, or mixed. Bond strength values were subjected to ANOVA and Tukey's HSD post-hoc test at a pre-determined significance level of 0.05.

Results

Experimental results and their statistical comparisons are presented in Figure 2. None of the photoactivation methods provided a totally intact margin: all methods demonstrated marginal gap formation. None of these values were significantly different, however. Thus, the photoactivation method had no significant influence on marginal adaptation.

Significant differences among curing methods were noted for internal adaptation. Pulse-delay and the soft-start methods resulted in the least gap formation. The highest internal gap formation values were found when using continuous light 700 and the intermittent light techniques that were equivalent. Specimens exposed to the low power, continuous method showed gap formations not significantly different from either of the two extremes just mentioned.

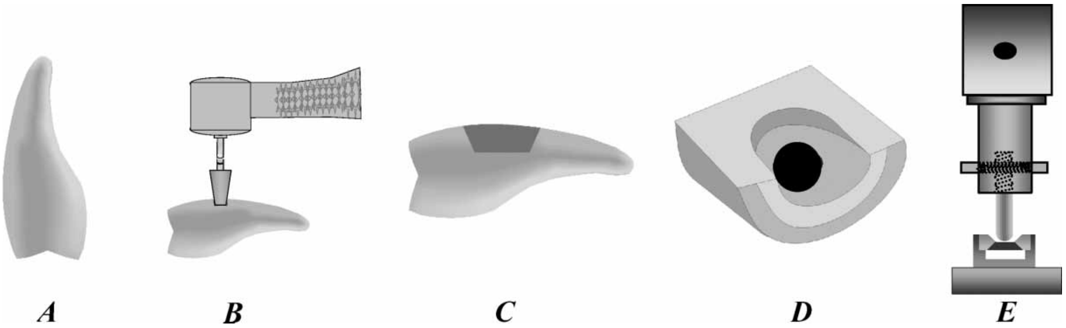

Figure 1. Schematic representation of the "push-out" test. A. Tooth crown. B. Preparation made using a specialized appliance. C. Lateral view of the restored specimen (2.0 mm in height, facial diameter 5.0 mm, and lingual diameter 4.0 mm). D. Selective removal of the lingual surface to expose the axial wall of the restoration. E. Lateral view of the specimen showing the direction of specimen push-out.

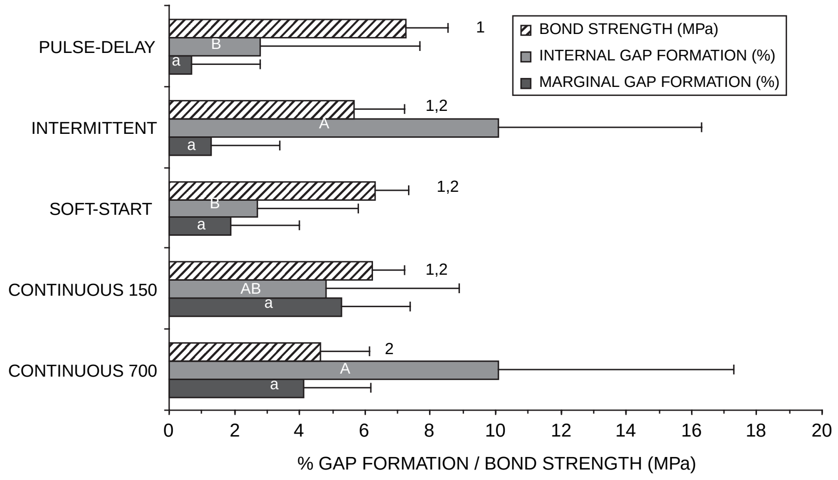

Figure 2. Results of parameter studies (mean (SD), horizontal bar +1 =standard deviation). Bars for parameter results containing similar codes (lower-case letter, upper-case letter, or number) are not statistically different within that parameter

The pulse-delay method showed the highest push-out strength value (7.3 ± 1.3 MPa) (Figure 2), but this was no different from methods using soft-start (6.3 ± 1.1 MPa), continuous low power density (6.2 ± 1.0 MPa), or intermittent light (5.7 ± 1.6 MPa). Continuous exposure using 700 mW/cm^2 resulted in the lowest strength value (4.6 ± 1.5 MPa), but was no different from the three methods mentioned previously. It can be seen from Figure 2 that there is an inverse relationship between internal adaptation values and push-out bond strength.

Observed failure mode proportions are show in Figure 3. For continuous light 700 and intermittent light, adhesive failure was the most observed mode (80%). In the continuous light 150, soft-start, and pulse-delay groups, 50% of failure mode was mixed failure.

Discussion

It is important to emphasize that each photocuring method used was adjusted to provide equivalent total energy to the composite: 14 J/cm^2. Thus, assuming equivalent and maximal conversion with each method, differences seen among the test parameters would reflect the rate and method that the curing light was applied, which would affect the rate of polymerization, the onset of vitrification, and thus the rate at which stress development occurred.

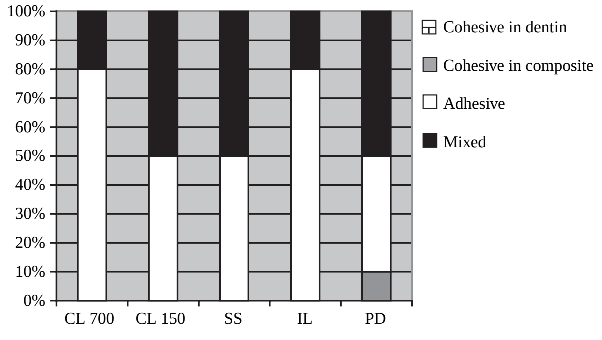

Figure 3. Percentage of failure mode in push-out bond strength test.

The push-out bond strength test is generally used to evaluate endodontic cements in the radicular canal space [27,28]. However, in the present study this test was adapted to evaluate strength of restorative composites in a simulated Class I preparation. The advantage of using this test is that the bond strength can be evaluated in a high C-factor environment, and all the bonded area submitted to mechanical force at the same time – the higher the C-factor, the higher the generated stress [3]. The C-factor used in the push-out test in the present study was 2.2 (a high stress condition).

An inverse relationship was observed between push-out bond strength and internal gap formation, i.e. the higher the bond strength, the lower the internal gap formation (Figure 2). This condition arises because internal gap formation lessens the total area that is bonded, and thus the lower the force required to dislodge the restoration. Also, both gap formation and bond strength are affected by stress generation during the polymerization process. As the photoactivation method can affect shrinkage stress generation [11,30], the internal gap formation and the bond strength will also be affected by the photoactivation method.

It is interesting to note that the photocuring method most used clinically (continuous light 700 photoactivation method) resulted in the lowest bond strength and the highest gap formation. Using this method, composite may undergo an immediate and rapid polymerization reaction, solidifying very quickly. Because of the high power density used, the initial exposure phase induces fast stress generation at the composite/dentin interface. The stress orientation to the adhesive interface during a rapid polymerization rate reduces the probability of bond preservation, and result in more internal gap formation. This assumption was confirmed by the high gap formation value of internal adaptation resulting from the continuous light 700 group: 10.1%. Therefore, a lower force is necessary to dislodge the restoration from specimens in this group during the push-out test. In addition, the predominant failure mode of this group was adhesive in nature (80%). This finding emphasizes weakening of the bond due to internal gaps.

The continuous, lower power density group (continuous light 150) showed no significant difference from the higher power density continuous light group, either with respect to marginal and internal adaptation or in push-out bond strength values. However, the continuous light 150 group presented a mixed failure mode predominantly. The reduced power density (150 mW/cm^2) could have enabled reduction of polymerization rate [1,14], resulting in slower stress development, increasing the probability of bond preservation. The internal gap formation for this group was 4.8%.

Intermittent light photoactivation is developed in cycles of intervals of light-on and light-off [9,16,30]. Theoretically, periods of light absence would extend the visco-elastic stage (vitrification) of the composite, resulting in reduced stress generation. Some studies [9,16,30] have pointed out the benefits of intermittent light on the photoactivation of composite. However, in these studies [9,16,30] the energy dose applied was lower than that of the other photoactivation methods. Reduction of energy dose leads to decreased shrinkage, and consequently, reduced shrinkage stress. The intermittent light method demonstrated similar bond strength, marginal and internal adaptation values as those obtained for the continuous light 700 group. However, there was no significant difference between this group and any of the other modulated photoactivation methods. Therefore, it can be stated that the light-off period was not long enough to cause a significant stress release and better results might have been obtained with a longer duration of light off. Other strobing durations should be evaluated in order to validate this concept.

Use of the soft-start technique had no effect on marginal adaptation. Other studies, too, have shown no improvement in marginal adaptation when this polymerization method was used [31–33]. It has been claimed that the optimum combination of dentin bonding and resin composite is more important than the irradiation method is with respect to marginal adaptation [32,33]. However, in the present study the evaluation of internal gap formation revealed that the soft-start technique did improve this parameter, a finding that may be attributed to the increased ability of composite to flow when polymerized at a reduced rate. Thus, development of the polymer network and cross-link formation is reduced, which may then allow the material to relieve stress through pre-vitrification plastic deformation, resulting in enhanced internal adaptation [16]. Ernst et al. [34] found that soft-start polymerization can significantly reduce polymerization stress.

The soft-start technique showed the intermediate value of push-out bond strength, which was not statistically different from the other photoactivation methods. The debonded specimens from this group presented the mixed failure predominantly, indicating partial preservation of the composite/dentin interface. This predominance of failure mode also correlates with what one would expect from the better internal marginal adaptation that was also observed.

The pulse-delay photoactivation method is a variation of the soft-start technique, providing a reduction of polymerization contraction stress without a significant reduction in degree of conversion [11]. This method has also been found to reduce marginal gap formation. In the present study, however, no significant difference was noted in marginal adaptation among all the experimental photocuring groups. Despite the absence of statistical differences, the marginal gap formation of the restorations cured using the pulse-delay method was the lowest (0.7%).

It has been stated that an initial power density of 150 mW/cm^2 is too high for polymerization shrinkage stress to be significantly reduced [11]. In the present study, however, even using 150 mW/cm^2 for 5 s resulted in a lowering of internal gap formation and significantly increased bond strength. This was attributed to reduction in shrinkage stress due to the slower curing rate.

A 3-min interval of light-off was selected as a delay between the two steps for pulse-delay in the present study. The objective of this extended light-off time was to allow the radicals that had been formed during the initial exposure to grow as a result of the "dark cure" phenomenon [19]. During this phase, no new radicals are formed, and further polymerization is the result of chain propagation of already existing structures. This growth occurs quite slowly, and is expected to slow the rate of further polymer growth while the material is still in a pre-vitrified state prior to the second exposure, which results in a great increase in the polymerization rate and rapid rise in modulus. Shrinkage prior to the acquisition of substantial modulus (vitrification) can be compensated for by molecular rearrangement of polymer chains (flow). Thus, introduction of the delay into the early portion of the light-curing routine may prolong the early low modulus phase, allowing the stress development to be relieved by polymer flow and deformation [11]. This situation was confirmed in this work, as the bond strength of this group was 7.3 MPa, statistically greater than that of the continuous light 700 group. The predominant failure mode of the pulse-delay group was mixed, due to the partial preservation of the adhesive interface.

In addition, fracture specimens from the pulse-delay group were the only ones to demonstrate a cohesive failure. The pulse-delay group also showed a significant reduction in internal gap formation (2.8%) when compared to the continuous light 700 group (control).

Additionally, it is important to point out some of the limitations of this study. The use of bovine teeth requires caution in interpretation of the results. However, the major problem with using bovine teeth occurs when different interactions between bonding systems and tooth substrate are compared. In this study, the interaction bonding system/tooth substrate was standardized, since the same bonding system and the same tooth location were used for all groups. The objective of this study was to evaluate the composite behavior under the confinement phase of the different photoactivation methods. In addition, the use of bovine incisors is supported by several authors [35–37].

Based on the results of the present study, the first hypothesis tested, namely that the alternative methods would significantly reduce marginal and internal gap formation and increase bond strength values relative to the control, can only be partially accepted. Modulated photoactivation methods demonstrated reduced internal gap formation and increased bond strength with respect to the use of the common, contemporary continuous exposure (control).

The second hypothesis tested, namely that there would be an inverse relationship between push-out bond strength values and marginal and internal gap formation, must also be only partially accepted. Marginal adaptation was not affected by any of the photoactivation methods and could not be related to bond strength or to internal gap formation. There was, however, an inverse relationship between bond strength and internal gap formation. This relationship could be confirmed in this study because both gap formation and bond strength were evaluated in high C-factor cavity preparations. Consequently, the polymerization shrinkage stress caused bond disruption, decreasing the bond strength values and increasing the internal gap formation, especially in the continuous light 700 group, in which the shrinkage stress is higher.

Based on the results of this research, the clinical application of modulated photoactivation methods should be encouraged. Although *in vitro* evidence suggests that use of these modulated techniques may offer significant clinical advantages, *in vivo* studies should be performed to validate these assumptions.

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