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Effect of Curing Mode on Microtensile Bond Strength to Dentin of Two Dual-cured Adhesive Systems in Combination with Resin Luting Cements for Indirect Restorations

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Clinical Relevance

The separate step of light curing the adhesive resin component of some fourth and fifth generation dual-cured adhesive systems may be eliminated prior to cementation of an indirect resin composite restoration without deterioration in microtensile bond strength.

SUMMARY

This study evaluated the microtensile bond strength (µTBS) of dual-cured adhesive systems when the different components were either light activated or left in the uncured state prior to cementation of an indirect composite restoration. Occlusal dentin surfaces of 40 human third molars were flattened. The teeth were randomly assigned to 8 groups (n=5) according to the dual-cured systems (bonding agents/resin cements) and curing modes: All Bond 2/Duolink (AB2-BISCO Inc) and Optibond Solo Plus Dual Cure/Nexus 2 (SOLO-Kerr). Resin cements were applied to pre-cured resin composite discs (2 mm thick/Z-250/3M ESPE), which were fixed to dentin surfaces containing adhesive resin in either cured (LP) or uncured states (SP). The restored teeth were light activated according to the manufacturers' instructions (LRC-XL3000/3M ESPE) or allowed to self-cure (SRC). The restored teeth were water-stored at 37°C for 24 hours. They were then both mesial-distally and buccal-lingually sectioned to obtain bonded specimens (1.2 mm²). Each specimen was tested in tension at a crosshead speed of 0.6 mm/minute until failure. Data (MPa (SD)) were analyzed by two-way ANOVA and Tukey's post-hoc test (*p*<.05). AB2/SP exhibited higher µTBS than AB2/LP (*p*=.00001); however, no significant differences were noted between SOLO/LP and SOLO/SP. Results suggested

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that dual-cured adhesive systems were as strong or even stronger when they were left in the uncured state prior to indirect resin composite cementation.

INTRODUCTION

Resin composite and ceramic inlay/onlay restorations are advocated as alternatives to metal restorations, because of increasing esthetic demand and advancements in adhesive dentistry.[1-2] The clinical success of composite and ceramic indirect restorations is attributed to the reliable bond between adhesive cementing systems (resin cements/bonding agents) and mineralized dental tissues.[3-4] However, as light intensity reaching the resin cement is strongly attenuated by either distance from the light source or from the absorbing characteristics through the indirect restorative material,[5] dual-cured resin materials have been developed.[6-7]

Dual-cured systems consist of a mixture of monomers and catalysts and are formulated so as not to depend solely on light activation for proper cure. Therefore, light activation of such systems prior to delivering an indirect restoration might not be necessary. This method of indirect restoration placement on uncured resin cement and the adhesive resin layer is usually recommended in an attempt to ensure an adequate marginal adaptation and to avoid incomplete seating of the restoration, which are the primary concerns of clinicians.

The pressure from luting composite during seating of an inlay/onlay may cause a collapse of the demineralized collagen fibers when the adhesive applied to dentin is not previously polymerized.[8] In addition, by evaluating *in vitro* occlusal wear, quantity of the remaining double bonds and hardness, some authors indicated that the chemical curing mechanism alone is less effective than the light-activated one when dual-cured restorative materials are used.[9-11] Based on this evidence, some manufacturers recommend light activation of dual-cured adhesive systems prior to applying resin cement and seating the restoration of the prepared tooth. However, the difference in bond strength between these two clinical techniques for cementation of indirect restorations when dual-cured adhesive systems are used has yet to be evaluated.

This study evaluated the microtensile bond strength (µTBS) of fourth and fifth generation dual-cured dentin bonding agents (adhesive resins) combined with their respective dual-cured resin cements when each is either allowed to self-cure or is exposed to light through a pre-cured disc of resin composite. In addition, the failure site morphology is classified and compared with respect to materials and curing mode type.

The research hypothesis was that independent light activation of both the resin adhesive and resin cement would result in significantly higher bond strengths than when either is allowed to self-cure only. In addition, it was expected that bond strengths would be greatest when the manufacturer's instructions are followed.

METHODS AND MATERIALS

Indirect Restorative Bonding Procedures

Forty freshly extracted, erupted human third molars stored in saturated thymol solution at 5°C for no more than 3 months, were used following a protocol approved by the Human Assurance Committee at The Medical College of Georgia (HAC #0403333). The teeth were transversally sectioned in the middle of the crown using a diamond blade (Series 15HC Diamond, #11-4244; Buehler Ltd, Lake Bluff, IL, USA) on an automated sectioning device (Isomet 2000; Buehler Ltd) under water irrigation, exposing areas of middle depth dentin. The exposed dentin surfaces were wet-polished by machine (Supermet Grinder, item #48-1581, Buehler Ltd) with 600-grit SiC paper (pn 810-281-PRM, Silicon Carbide PSA Discs, Leco Corp, St Joseph, MI, USA) to create a flat surface with standard smear layer formation before bonding with the adhesive systems. The prepared teeth were randomly divided into 8 groups (n=5 specimens per group).

Commercial fourth- and fifth-generation dual-cured dentin adhesive systems were used (Table 1). The corresponding dual-cured resin cements from each manufacturer were also applied (Table 1). Forty light-activated resin composite discs (2-mm thick and 10 mm in diameter—A2 shade—Z250, lot #5LB; 3M/ESPE, St Paul, MN, USA) were prepared to simulate overlying laboratory-processed resin composite restorations. The surface of each pre-cured resin disc that was to be bonded was sandblasted with 50 µm aluminum oxide particles (lot #51116150, micron white, Danville Engineering Inc, San Ramon, CA, USA) for 10 seconds (air pressure: 80 psi; distance from the tip: 1.5 cm) (Comco MB 1002; COMCO Inc, Burbank, CA, USA). All adhesive systems and resin cements were manipulated and applied to the dentin surfaces according to the manufacturers' instructions (control): light activation (20 seconds, light intensity: 550—630 mW/cm^2, XL3000, sn #202149; 3M/ ESPE) of the Primer A and B mixture of All Bond 2 (BISCO Inc, Schaumburg, IL, USA) as well as the mixture of Optibond Solo Plus Dual Cure (Kerr Corp, Orange, CA, USA) and the activator component prior to placement of the respective resin cements (Table 1). For the experimental groups, all adhesive systems were applied and left in the uncured state, relying totally on any self-curing mechanism.

The mixed resin cement pastes were applied to the pre-cured composite disc following manufacturers' instructions, and the disc was positioned and fixed to the adhesive-coated dentin surface under load of 500 g for five allowed to self-cure. When the cementing materials were light-activated through the pre-cured composite disc, the curing unit tip was positioned against the com-

Table 1: *Composition of the Dual-Cured Adhesive Systems Used in This Study*

Product (Manufacturer)	Composition	Batch #	Manufacturer's Instructions
All Bond 2 (BISCO Inc)	*Primer A*: acetone; ethanol; Na-N-tolylglycine glycidylmethacrylate. *Primer B*: acetone; ethanol; biphenyl dimethacrylate. *Pre-Bond Resin*: Bis-GMA, TEGDMA; benzoyl peroxide; BHT.	0500003574 0500003579 0500004345	Mix primers A and B. Apply 5 consecutive coats to dentin. Dry all surfaces for 5-6 seconds with an air syringe. Light-cure 20 seconds.
Duolink (BISCO Inc)	*Base*: Bis-GMA; TEGDMA; glass filler; urethane dimethacrylate. *Catalyst*: Bis-GMA; TEGDMA; glass filler	0500003751	Apply thin layer of Pre-Bond Resin immediately prior to cementation. Air thin. Do not light-cure.
Optibond Solo Plus Dual Cure (Kerr)	*Adhesive Resin*: ethyl alcohol; Bis-GMA; HEMA; GPDM; photoinitiators; barium aluminoborosilicate glass; fumed silica (silicon dioxide); sodium hexafluorosilicate. *Activator*: ethyl alcohol; alkyl dimethacrylate resins; benzene sulfinic acid sodium salt.	428904 428260	Dispense one drop of Optibond Solo Plus and Optibond Solo Activator into a disposable mixing well. Mix for 3 seconds. Apply mixture to dentin with a light brushing for 15 seconds to cover dentin surface. Lightly air thin for 3 seconds. Light-cure for 20 seconds.
Nexus 2 (Kerr)	Monomers of methacrylic acid esters, Ba–Al–borosilicate glass, chemical and photoinitiators.	Base: 423638 Catalyst: 423975	

PENTA, dipentaerythritol penta acrylate monophosphate: HEMA, 2-Hydroxyethyl Methacrylate; BISGMA, bisphenol-a glycidylmethacrylate
TEGDMA: triethileneglycol dimethacrylate: BHT, butylated hydroxytoluene; GPDM, glycerophosphate dimethacrylate.

posite disc, and each sample was exposed for 40 seconds (XL 3000, 3M/ESPE). A 2-mm thick block of self-curing resin composite (lot #0500006449, shade A3/A3.5, Bisfil 2B, BISCO Inc) was then added to the untreated, cured composite surface to allow easier specimen manipulation while the mechanical test was performed. For groups where resin cements were self-cured, the block of self-curing resin composite was applied to the composite disc only after the time stipulated for the cement's self-cure reaction to complete.

Microtensile Bond Strength Test (μTBS)

The restored teeth were stored in distilled, deionized water at 37°C for 24 hours and vertically, serially sectioned into several 0.8-mm thick slabs using the same cutting instrument previously mentioned. Each slab was further sectioned to produce bonded sticks of approximately 1.2 mm^2. Each bonded stick was attached to the grips of a microtensile testing jig (BISCO Inc) with cyanoacrylate cement (Zapit, Dental Ventures of America Inc, Corona, CA, USA) and tested in tension on a universal testing machine (Vitrodyne V1000 Universal Tester, Chatillon, Greensboro, NC, USA) at a crosshead speed of 0.6 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 fracture was measured to the nearest 0.01 mm with a digital micrometer (Series 406; Mitutoyo America Corp, Aurora, IL, USA). Specimen cross-sectional areas were calculated in order to present μTBS data in units of stress: MPa.

Statistical Analysis

A 2-way analysis of variance (ANOVA) (effect of adhesive resin curing mode, effect of resin cement curing mode) was performed for each dual-cured adhesive system, because the purpose of this study was not to compare product strengths, but to evaluate curing mode techniques. Tukey's post-hoc test was used to detect pair-wise differences within a bonding system. All statistical testing was performed at a pre-set alpha of 0.05.

Failure Pattern Analysis

Fractured surfaces of tested specimens were allowed to dry overnight at 37°C. The surfaces were sputter-coated with gold (Model EMS-76M, Fullan Corp, NY, USA) and observed under a scanning electron microscope (XL-30, Philips. Hillsboro, OR, USA). Failure patterns were classified as follows at the resin cement adhesive: cohesive along the resin cement-adhesive layer interface, cohesive within the resin cement, adhesive along the pre-cured composite overlay-resin cement interface, adhesive either within or at the top of the hybrid layer and adhesive resin layers, and mixed when simultaneously exhibiting remnants of both hybrid layer and resin cement.[12]

RESULTS

Microtensile Bond Strength Test

The μTBS results are displayed in Table 2. When All Bond 2 was applied, light activation of the primer resulted in more than 50% lower μTBS values than

Table 2: *µTBS of the 4th and 5th Generation Dual-cured Adhesive Systems*

Bonding System Generation (product)	Resin Cement Curing Mode	Adhesive Resin Curing Mode	
		Light-activated	Self-cured
4th Generation	Light (LRC)	14.6 (2.2)Aa	36.2 (5.6)Ab
(All Bond 2)	Self (SRC)	13.9 (1.8)Aa	37.8 (8.0)Ab
5th Generation	Light (LRC)	32.9 (4.9)Aa	34.1 (7.8)Aa
(Optibond Solo Plus Dual Cure)	Self (SRC)	26.3 (6.9)Ba	23.7 (3.8)Ba

Groups having similar letters (upper case = column; lower case = row) are not significantly different.

when the primer was not light-activated before the resin cement was applied ($p<.0001$). The mode of resin cement cure did not affect µTBS regardless of the mode of primer layer cure.

On the other hand, when Optibond Solo Plus Dual Cure was used, no significant difference in µTBS was observed when the adhesive resin layer was light activated or left in the uncured state. However, the mode of resin cement cure affected the tensile bond strength regardless of the mode of cure of the adhesive resin ($p=0.0036$): 24.7% (for light-activated adhesive resin) and 46.4% (for self-cured resin) higher µTBS when the resin cement was light-activated than when it was allowed to self-cure.

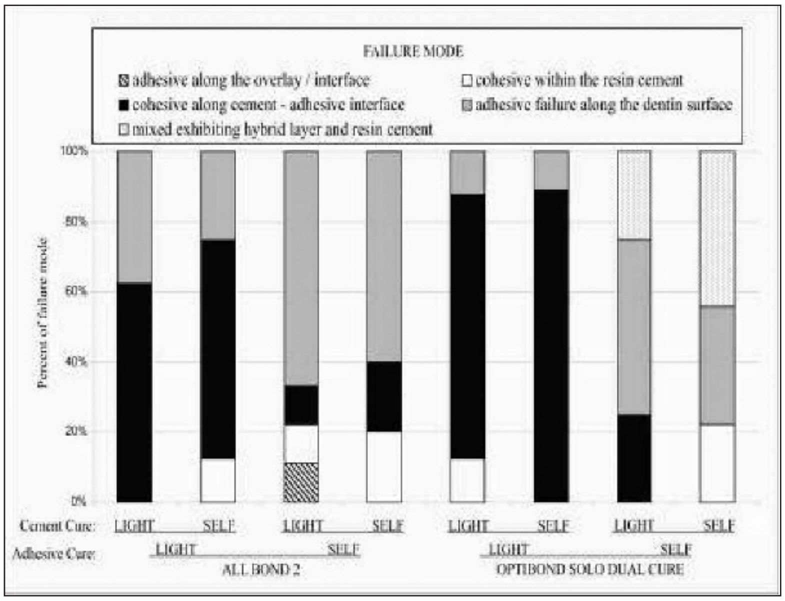

Figure 1. *Proportional prevalence (%) of failure patterns for all experimental groups.*

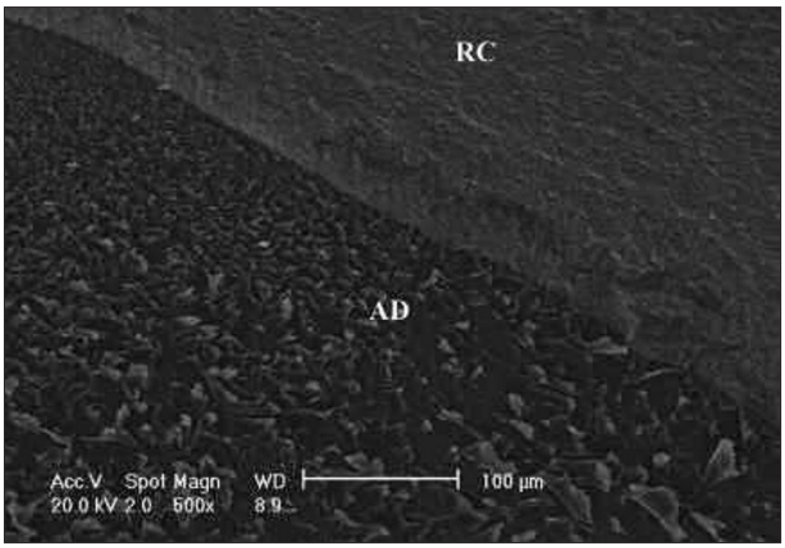

Figure 2. *Fractured specimen using All Bond 2 when primer was light activated, exhibiting failure pattern classified as cohesive along the adhesive layer (AD) and resin cement (RC) interface (magnification 500x). This was the most predominant failure pattern observed when All Bond 2 was light activated and the resin cement was either light- or self-cured and when Optibond Solo Plus Dual Cure was light activated, but its resin cement was self-cured only.*

Failure Pattern Analysis

Figure 1 shows the proportional prevalence (%) of the failure patterns in all experimental groups. For All Bond 2, the most predominant failure pattern was at the cement-adhesive interface when the primer was light activated (Figure 2). However, adhesive failure occurring either within or at the top of the hybrid layer and adhesive resin layers was the most predominant failure pattern when the primer was not light activated separately (Figure 3). A higher incidence of cohesive failure within the resin cement was observed when the resin cement was allowed to self-cure rather than when it was light-activated (Figure 1).

An adhesive failure mode located either within or at the top of the hybrid layer and adhesive resin layers was also the most predominant failure pattern noted for Optibond Solo Plus Dual Cure when the adhesive layer was not independently light-activated and the resin cement was light-activated (Figure 3). Cohesive failure along the cement-adhesive interface was predominantly observed when the adhesive layer was light-activated (Figure 4). A mixed failure between resin cement and dentin surfaces was most commonly observed when both adhesive layer and resin cement were allowed to self-cure (Figure 5).

DISCUSSION

The results of this study demonstrate that the method of curing mode used when cementing indirect composite restorations may affect the tensile bond strength of indirect restorations, depending on the cementing system used.

Therefore, the research hypothesis that light activation of dual-cured adhesive systems would result in significantly higher bond strengths than when they are allowed to self-cure only was rejected. On the other hand, the research hypothesis that light activation of dual-cured resin cements would increase µTBS when compared to self-cured groups was accepted for Optibond Solo/Nexus2 but was rejected for All Bond 2/Duolink, regardless of the mode of cure established for the dual-cured adhesive systems.

Surprisingly, when used according to the manufacturer's instructions (primer light-activated for 20 seconds), All Bond 2 exhibited lower µTBS than when the primer was left in the uncured state before seating the indirect restoration. This difference was unexpected, since optimal primer polymerization (and thus physical properties) is expected when light exposing the resin adhesive directly. The failure pattern observed when the primer was light-activated and the resin cement was either light-activated or allowed to self-cure was predominantly located at the interface between the adhesive resin layer and the resin cement (Figure 2). A similar failure pattern was observed by Mak and others[12] when All Bond 2 was applied to dentin and an indirect composite onlay was cemented with resin cement. According to the authors, the failure pattern observed may be attributed to the inclusion of a high concentration of butylated hydroxytoluene (BHT), a polymerization inhibitor present in the adhesive resin (Pre-Bond) for the purpose of controlling the accelerated rate of cure of the resin cement caused by the presence of tertiary amine-based resin monomers in Primer A. The reduction in reaction speed by chemical inhibition occurs as free radicals are terminated by reacting with phenolic hydrogen of the BHT molecule.[13] Therefore, it is possible that, when Pre-Bond was combined with resin cement, the decrease in available free radicals may have impaired the polymerization reaction of Duolink even when the resin cement was light-activated.

Although the manufacturer recommends that the primer must be light-activated for 20 seconds, the µTBB of All Bond 2 without primer light activation was significantly higher than that obtained when the manufacturer's instructions were followed. The possible explanation for this finding may be related to the mixture among the primer and adhesive resin components within the hybrid layer when Pre-Bond is applied to the primed surface. Composed of a high concentration of hydrophobic monomers, such as Bis-GMA (Table 1), Pre-Bond adhesive resin may create a hybrid layer with a high concentration of hydrophobic monomers and, consequently, lower hydrophilicity,[14-16] thus improving its mechanical properties.[17-18] Moreover, dual-curing mechanisms within the hybrid

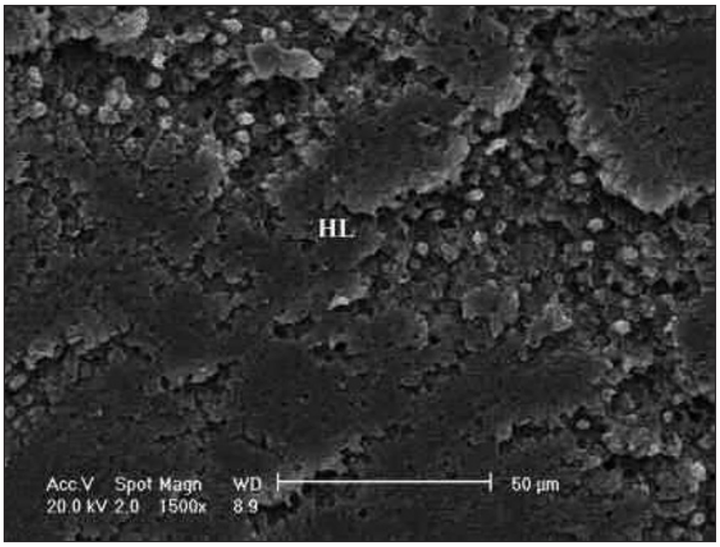

Figure 3. *Fracture located within the hybrid layer (HL) was the most predominant failure pattern for All Bond 2 when the primer was allowed to self-cure, and for Optibond Solo Plus Dual Cure, when the adhesive layer was left in the uncured state and the resin cement was light activated (magnification 1500x).*

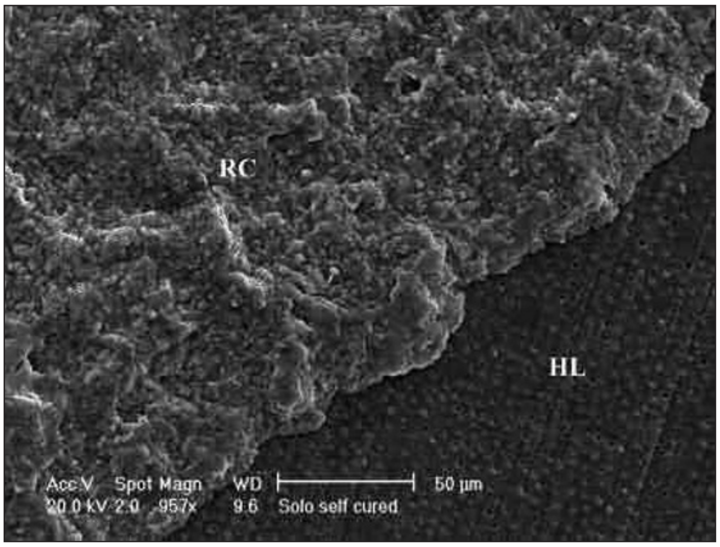

Figure 4. *Fractured specimen exhibiting cohesive failure pattern along the adhesive layer (AD) and resin cement (RC) interface (magnification 150x). This was the most predominant failure pattern observed when Optibond Solo Plus Dual-Cure was light activated and its resin cement was self-cured.*

layer may have contributed to the higher µTBS when Pre-Bond resin infiltrated the primed dentin surface. Considering that All Bond 2 Primer A contains a tertiary amine as a component for the self-curing reaction,[12] and Pre-Bond resin has benzoyl peroxide, it is possible that free-radicals within the hybrid layer might have been created not only from light activation, but also from the self-curing redox reaction. As a consequence, a high content of free radicals may be available for proper polymerization reaction to occur, even in the presence of a high amount of BHT in Pre-

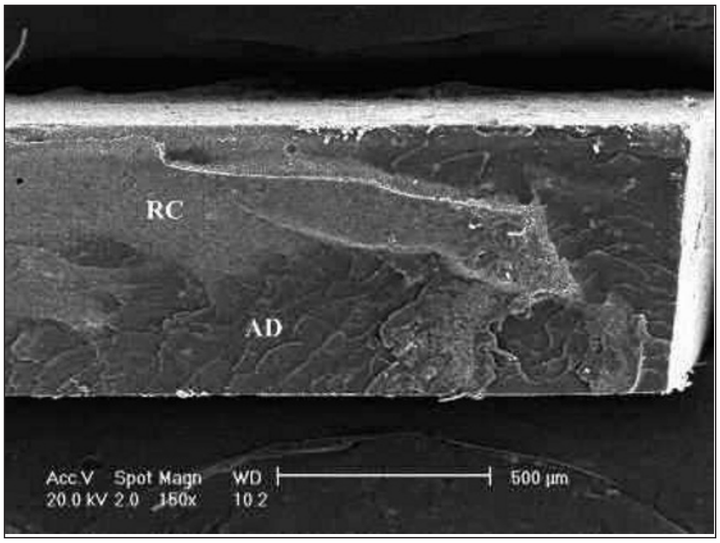

Figure 5. *Representative SEM photomicrograph of mixed failure pattern exhibiting resin cement (RC) and hybridized dentin surface (HL) (magnification 954x). This failure pattern was mostly observed when both the adhesive resin and resin cement of Optibond Solo Plus Dual Cure/Nexus 2 were allowed to self-cure.*

Bond resin. Once the inhibitor is completely consumed, polymerization reaction of the resin cement will proceed.

The research hypothesis related to the effect of the curing mode of dual-cured adhesive systems on µTBS proposed in this study was not accepted for Optibond Solo Plus Dual Cure. No differences in µTBS were observed when the adhesive system was either light activated or allowed to self-cure before indirect resin composite cementation. One possible explanation for this finding may be related to the presence and effectiveness of a co-initiator component in the adhesive system. When resin cement was applied to the uncured adhesive layer, the adhesive layer was replaced by a new combined layer composed of a mixture of resin cement and adhesive resin. Without the presence of a co-initiator, such as benzene sulfinic acid sodium salt, the tertiary amines from the peroxide-amine component can react with acidic monomers to form a charge transfer complex (CT complex)[21] and loose their ability as reducing agents in redox reaction,[19-22] and a poor polymerization reaction can be expected from the combined adhesive/resin cement layer. However, when a separate co-initiator component is added to the adhesive resin, it reacts with the acidic monomers to form a phenyl-free radical against the CT complex. For this reason, it is speculated that the combined adhesive/resin cement layer was allowed to self-cure properly when the co-initiator was included in the composition of Optibond Solo Dual Cure.

Other factors may have contributed to the high µTBS observed when Optibond Solo was not light activated. When compared to the adhesive layer alone, the combined adhesive/cement layer would have higher filler content and more hydrophobic monomers, which would provide improved mechanical properties,[23] lower shrinkage[24] and less susceptibility to hydrolytic degradation.[25] In addition, it is possible that the combined adhesive/cement layer is able to penetrate the entrance of dentinal tubules and increase the strength of the dentin bonding interface. Further studies are necessary to evaluate the micromorphology of the dentin bonding interface created by indirect bonding procedures without light-activating the adhesive layer.

The effects of the resin cement curing modes on µTBS were also found to be material-dependent. For All Bond 2, the curing mode of Duolink did not affect the tensile bond strength, while lower µTBS was observed when Nexus 2 was allowed to self-cure after Optibond Solo was either light-activated or left in the uncured state. Evidence that the co-initiator of Optibond Solo was effective when Nexus 2 was applied to the light-activated adhesive layer may confirm that the self-curing mechanism by itself is ineffective in providing reliable mechanical properties to resin cements as previously reported.[9-11,26-27] This hypothesis was confirmed when fracture analysis of µTBS specimens revealed the failure pattern predominantly located at the bottom of the resin cement layer (Figure 4).

Mixed failure exhibiting both the hybrid layer and regions with resin cement was the most predominant failure pattern observed when both adhesive resin and resin cement were allowed to self-cure (Figure 5). This finding may indicate that the self-curing components were not able to provide high cohesive strength to adhesive resin within the hybrid layer when the activating light is not available. This evidence is a matter of concern, because weakly polymerized unfilled resins are more susceptible to an accelerated degradation process.[28-29] Further investigation is needed to evaluate efficacy of the self-curing mechanism of dual-cured adhesive systems and the influence of the self-cure reaction of the adhesive/cement layer on the polymerization of these adhesive resins.

Inclusion of an adhesive system without any self-curing or co-initiator components in this study could provide some indirect evidence regarding the effects of these self-curing components on the mechanical properties of the bonding interface. However, this study aimed only to evaluate how effective some specific dual-cured adhesive/resin cement systems are when indirect composite restorations are bonded to dentin. Thus, there is still a lack of information about the effectiveness and limitations of dual-cured bonding agents when they are used for indirect porcelain/composite restorations.

This study evaluated the effect of an alternative technique for indirect resin composite cementation when one-fourth and one-fifth generation dual-cured adhesive systems were used. As a comparison of products was deemed unimportant, only a single two-way ANOVA (mode of cure of resin cement factor; mode of cure of the adhesive systems factor) was performed for each product instead of a three-way ANOVA, including the products together. According to study results, the alternative technique of allowing all components to self-cure provided µTBS equivalent to or significantly greater than that observed when the adhesive systems were light-activated. Therefore, this alternative technique for the fourth- and fifth-generation dual-cured adhesive systems may be a reliable option even in the worst clinical conditions where light exposure is totally compromised. However, further studies are necessary to confirm the effectiveness of other fourth- and fifth-generation dual-cured adhesive systems when no light exposure is available.

CONCLUSIONS

Within the limitations of this study, the following conclusions were observed:

1. The research hypothesis that independent light activation of both resin adhesive and resin cement would result in significantly higher bond strengths than when either is allowed to self-cure only was rejected for the fourth-generation dual-cured adhesive system evaluated. However, it was accepted for the fifth product only when the independent light-activation of both resin adhesive and resin cement values were compared to those obtained when the resin cement was allowed to self-cure, regardless of the curing mode of the adhesive resin.
2. When the manufacturers' instructions were followed, the bond strength values were either similar to or lower than those obtained when the alternative method of using self-curing for both adhesive and cement systems when delivering an indirect restoration.

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