

The Effect of the Presence and Presentation Mode of Co-Initiators on the Microtensile Bond Strength of Dual-Cured Adhesive Systems Used in Indirect Restorations

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Clinical Relevance

The presentation mode of co-initiators added to bonding agents may affect the bond strength of indirect composite restorations to dentin.

SUMMARY

This study compared the microtensile bond strength (μ TBS) of indirect resin composite restorations to dentin when fifth-generation

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adhesive systems were either light-activated or left in the uncured state prior to cementation. The systems used in this study were Prime&Bond NT (NT-Dentsply) and Excite (EX-Ivoclar-Vivadent) or their dual-cured versions containing co-initiators with different presentation modes (as solution or salts) Prime&Bond NT Dual-cure (NTD-Dentsply) and Excite DSC (DSC-Ivoclar Vivadent). The bonding agents were applied to the flattened occlusal dentin surfaces of 40 human third molars according to the manufacturers' instructions and were light-activated (XL3000/3M ESPE) for 10 seconds (LP) or left in the uncured state (SP). The respective resin cements Calibra (Dentsply Caulk) and Variolink II (Ivoclar Vivadent) were applied to pre-cured resin composite discs (2 mm thick/Z-250/3M ESPE) that were fixed to the bonded dentin surfaces. The restored teeth were light-activated according to the manufacturers' instructions for five minutes after cementation and water-stored at 37°C for 24 hours. The teeth were then both mesial-distally and buccal-lingually sectioned to obtain bonded specimens (0.8 mm²). Each speci-

men was tested in tension at a crosshead speed of 0.5 mm/minute until failure. The data (MPa [SD]) were analyzed by 3-way ANOVA and Tukey's post-hoc test ($p < .05$). The results showed that the presence of a co-initiator decreased μ TBS for NT, while no significant difference in μ TBS was noted between EX and DSC, regardless of the curing mode. All SP groups exhibited lower μ TBS than the LP groups. The results suggest that co-initiators presented as a solution may decrease μ TBS to dentin of the evaluated adhesive systems in indirect resin composite restorations. Light-activation of the adhesive layer prior to indirect cementation was crucial for higher μ TBS on dentin.

INTRODUCTION

The clinical success of composite and ceramic indirect restorations is related to the effectiveness of cementing systems (combination of dentin bonding agents and dual-cured resin cements) in promoting proper bonding to teeth.^{1,2} The luting procedure for indirect restorations most commonly recommended by manufacturers is based on light-activation of the adhesive resin before indirect restoration cementation. However, some studies have demonstrated that a thick, cured adhesive layer can affect full seating and, as a consequence, the marginal adaptation of indirect restorations.³⁻⁵ In an attempt to avoid such an issue, another clinical approach was developed in which the dentin bonding agent is left in the uncured state prior to application of the resin cement. This technique allows for maximum restoration seating⁴ and might create a combined adhesive layer composed of the mixture of bonding agent and resin cement.

Regardless of the clinical approach used for luting indirect restorations to teeth, it has been reported that acidic resin monomers from two-step total-etch and self-etch adhesives may impair the polymerization of dual-cured cements and composites that are initiated via peroxide-amine binary redox catalysts.⁶⁻⁷ As a consequence, low bond strength values are reported when light activation of the dentin bonding agent is not performed.^{8,9} In order to avoid this chemical incompatibility, manufacturers have added co-initiators, such as aryl sulfinic acid salts, organoboron compounds and barbituric acid/cupric chloride to the so-called dual-cured adhesive systems.¹⁰ When such systems are used, the co-initiators react with the acidic resin monomers to produce either phenyl or benzenesulfonyl-free radicals that initiate the reaction of dual-cured resin cements when light from the curing unit is not available.¹⁰

As most co-initiators consist of salts, they are usually mixed in organic solvents, such as acetone or ethanol, and are presented in single bottles. The solution composed of these salts and organic solvents is mixed with

the bonding agent, and the mixture is applied to dentin according to the manufacturer's instructions, following the same procedures recommended for bonding agents without co-initiators. Co-initiator salts can be also impregnated onto specific brushes provided by the manufacturers, so that no organic solvents are necessary to act as the vehicle. However, little information is available regarding the effect of the presence of co-initiators and their presentation mode (salts or solution) on the bond strength of adhesive systems to dentin when indirect restorations are cemented onto teeth.

Therefore, this study evaluated the effects of adding co-initiators with different presentation modes to adhesive systems on the microtensile bond strength (TBS) of indirect resin composite restorations to dentin. Also investigated were the effects of the clinical approach (the option of light-activating the adhesive layer prior to applying the resin cement or leaving the adhesive layer in the uncured state) for cementing indirect restorations when co-initiators were either added or avoided.

The research hypotheses of the current study were: 1) the addition of co-initiator to the bonding agent provides higher bond strength, regardless of the curing mode of the adhesive layer and 2) the presentation mode of the co-initiator (as a salt or diluted in organic solvent) is not related to the bond strength of indirect restorations, regardless of the curing mode of the adhesive layer.

METHODS AND MATERIALS

Indirect Restorative Bonding Procedures

Forty freshly extracted erupted human third molars, stored in saturated thymol solution at 5°C for no longer than three months, were used following a protocol approved by the Human Assurance Committee at Piracicaba School of Dentistry (HAC #054/2006). The teeth were transversally sectioned in the middle of the crown using a diamond blade (Buehler Ltd, Lake Bluff, IL, USA) on an automated sectioning device (Isomet 1000; Buehler Ltd) under water irrigation, exposing areas of middle depth dentin. The exposed dentin surfaces were wet-polished by machine (APL 4, Arotec SA, Cotia, SP, Brazil) with 600-grit SiC paper (3M do Brasil, Jacarei, SP, Brazil) to create a flat surface with a standard smear layer before being bonded with the adhesive systems. The prepared teeth were randomly divided into eight groups ($n=5$ specimens per group).

The commercial fifth-generation dual-cured dentin adhesive systems Prime & Bond NT (Dentsply Caulk, Milford, DE, USA) and Excite (Ivoclar Vivadent, Schaan, Liechtenstein), as well as their respective dual-cured versions Prime & Bond NT Dual-Cure (which contains co-initiator presented as a solution in a separate bottle) and Excite DSC (which presents the co-ini-

tiator as salt impregnated in brushes) were used (Tables 1 and 2). The corresponding dual-cured resin cements from each manufacturer were also applied (Table 3). Forty pre-cured 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 to be bonded was sandblasted with 50 µm aluminum oxide particles (Microetcher II, Danville Engineering Inc, San Ramon, CA, USA) for 10 seconds (air pressure: 80 psi; distance from the tip: 1.5 cm). All the adhesive systems and resin cements were manipulated and applied to the dentin surfaces according to the manufacturers' instructions and either light activated for 10 seconds (light intensity: 550—630 mW/cm², XL3000; 3M ESPE) or left in the uncured state prior to the resin cement application.

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 a load of 500 g for five minutes, during which the resin cement was allowed to self-cure. Then, the curing unit tip (diameter of light guide tip: 8 mm, XL 3000, 3M ESPE) was positioned against the composite disc and each cementing material was light-activated through the pre-cured composite disc for 40 seconds. A 2-mm thick block of self-curing resin composite (shade A3/A3.5, Bisfil 2B, BISCO Inc, Schaumburg, IL, USA) was then added to the untreated, cured composite surface to allow easier specimen manipulation while the mechanical test was performed.

Table 1: Brand, Composition, Batch # and Manufacturers' Instructions of the 5 th Generation Dual-cured Adhesive Systems Used		
Product (Manufacturer)	Composition Manufacturer Supplied (Batch #)	Manufacturer's Instructions and Exceptions
Excite (Ivoclar-Vivadent)	<i>Adhesive Resin:</i> Alcohol, phosphonic acid acrylate, HEMA, SiO ₂ , initiators and stabilizers, Dimethacrylates (G00049)	Saturate enamel and dentin with a generous amount of Excite using a Vivadent applicator or a comparable application aid. Gently agitate the adhesive onto all prepared dentin surfaces for at least 10 seconds. This may be achieved using a gentle, clean, dry stream of air for 1-3 seconds. Cure Excite with halogen, plasma arc or laser polymerization lights.
Excite DSC (Ivoclar-Vivadent)	<i>Adhesive Resin:</i> Alcohol, phosphonic acid acrylate, HEMA, SiO ₂ , initiators and stabilizers, Dimethacrylates <i>Activator:</i> Aromatic Sodium Sulvinat salt (self-cure initiator) (H17469)	Gently agitate the adhesive onto all prepared dentin surfaces for at least 10 seconds. This may be achieved using a gentle, clean, dry stream of air for 1-3 seconds. If a pre-cure technique is preferred, Excite may be cured with halogen lights. Avoid pooling, as this may compromise the accuracy of fit of the definitive restoration.
HEMA: 2-Hydroxyethyl methacrylate; SiO ₂ : Silicon Oxide.		

Table 2: Brand, Composition, Batch # and Manufacturers' Instructions of the 5 th Generation Dual-cured Adhesive Systems Used		
Product (Manufacturer)	Composition Manufacturer Supplied (Batch #)	Manufacturer's Instructions and Exceptions
Prime & Bond NT (Dentsply Caulk)	<i>Adhesive Resin:</i> Acetone, Urethane dimethacrylate resin, Dipentaerythritol pentaacrylate phosphate, Polymerizable dimethacrylate resins, Polymerizable trimethacrylate resins (050413)	Place 1-2 drops of the adhesive into a mixing well. Using the disposable brush supplied, immediately apply adhesive to thoroughly wet all the tooth surfaces; remove excess solvent by gently drying with a dental syringe for at least five seconds. Surface should have a uniform glossy appearance. Cure mixed adhesive/activator for 10 seconds using a curing light unit.
Prime & Bond NT Dual Cure (Dentsply Caulk)	<i>Adhesive Resin:</i> Acetone, Urethane dimethacrylate resin, Dipentaerythritol pentaacrylate phosphate, Polymerizable dimethacrylate resins (050413) <i>Activator:</i> Aromatic Sodium Sulfinat (Self-cure initiator), Acetone, Ethanol (041110)	Place 1-2 drops of the adhesive and an equal number of drops of Self-Cure Activator into a mixing well; Mix contents for 1-2 seconds with a clean, unused brush tip; Using the disposable brush supplied, immediately apply mixed adhesive/activator to thoroughly wet all the tooth surfaces. These surfaces should remain fully wet for 20 seconds and may necessitate additional applications of mixed adhesive/activator. Remove excess solvent by gently drying with a dental syringe for at least five seconds. Surface should have a uniform glossy appearance. Cure mixed adhesive/activator for 10 seconds using a curing light unit.
HEMA: 2-Hydroxyethyl methacrylate.		

Table 3: Brand, Composition and Batch # of the Dual-cured Resin Cements Used

Product (Manufacturer)	Composition	Batch #
Calibra (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, tertiary amines. Catalyst paste: Barium boron, fluoroalumino silicate glass, Bis-GMA resin, Polymerizable dimethacrylate resin, Hydrophobic Amorphous Fumed Silica, Titanium Dioxide, Benzoyl Peroxide.	Base: 0504111 Catalyst: 0505121
Variolink (Ivoclar-Vivadent)	Paste of dimethacrylates, inorganic fillers, ytterbiumtrifluoride, initiators, stabilizers and pigments Bis-GMA; Triethylene glycoldimethacrylate; Urethanedimethacrylate; Benzoylperoxide.	Base: H23580 Catalyst: H20924
TEGDMA: Triethylene glycol dimethacrylate, Bis-GMA: Bisphenol A diglycidyl ether methacrylate, UDMA: Urethane dimethacrylate, HDDMA: 1,6-hexanediol dimethacrylate.		

Table 4: MTBS Means of Cementing Systems With or Without Co-initiators After Different Curing Modes of the Adhesive Layer

		Co-initiator	Without Co-initiator
Light-activated	Prime & Bond NT	23.87 (9.33)Bb	32.41 (4.55)Aa
	Excite	31.49 (8.90)Aa	25.32 (5.86) Ab
Self-cured	Prime & Bond NT	13.06 (5.78)Bb	24.37 (9.55) Aa
	Excite	19.79 (9.940)Aa	16.48 (5.65) Ab
Significant differences are represented by different letters (Capital letter—row; lower case letters—column) when three-way ANOVA and Tukey's post-hoc test were performed. Comparison between light-activated and self-cured groups is not displayed in this table. All light-activated groups show higher MTBS than the self-cured groups ($p=0.003$).			

Microtensile Bond Strength Test (μ TBS)

The restored teeth were stored in distilled, deionized water at 37°C for 24 hours and serially sectioned in a vertical dimension into approximately 25 0.8-mm thick slabs using the same cutting instrument previously mentioned. Each slab was further sectioned to produce bonded sticks approximately 0.8 mm². Six beams were randomly selected from the middle of each restored tooth and fixed to the grips of a testing device using cyanoacrylate (Zapit, Dental Ventures of America Inc, Corona, CA, USA). The specimens were then tested in tension in a universal testing machine (4411; Instron Co, Canton, MA, USA) at 0.5 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 with a digital micrometer (Starret 727-6/150, Starret, Itu, Sp, Brazil). The specimen cross-sectional areas were calculated in order to present μ TBS data in units of stress: MPa. The μ TBS of each tooth was the result of an average obtained from the values of the six tested beams, because such beams should not be considered separate experimental units.¹¹ Therefore, the use of beams located at the middle of the dentin surface and the use of an average to represent the μ TBS of one tooth contributed to reducing the effects of intra-tooth variability in the results, as previously demonstrated.¹¹⁻¹³

Statistical Analysis

A three-way analysis of variance (ANOVA) (effect of adhesive brand, effect of the presence of co-initiator, effect of adhesive activation) and Tukey's post-hoc test were performed to detect pair-wise differences among the groups. All statistical testing was performed at a pre-set alpha of 0.05.

Failure Pattern Analysis

The fractured surfaces of the tested specimens were allowed to dry overnight at 37°C. The surfaces were sputter-coated with gold (SDC 050, Baltec, Vaduz, Liechtenstein) and observed using a scanning electron microscope (VP-435, Leo, Cambridge, United Kingdom). Failure patterns were classified as follows: adhesive along the resin cement-adhesive interface, cohesive within the resin cement, adhesive along the dentin surface and mixed when simultaneously exhibiting remnants of both the hybrid layer and resin cement.¹⁴

RESULTS

Microtensile Bond Strength Test

The μ TBS results are displayed in Table 4. The ANOVA analysis detected statistical significance for the "adhesive activation" factor, and the Tukey test demonstrated that all cementing systems exhibited lower bond strength when the adhesive layer was left in the uncured state prior to the resin cement application

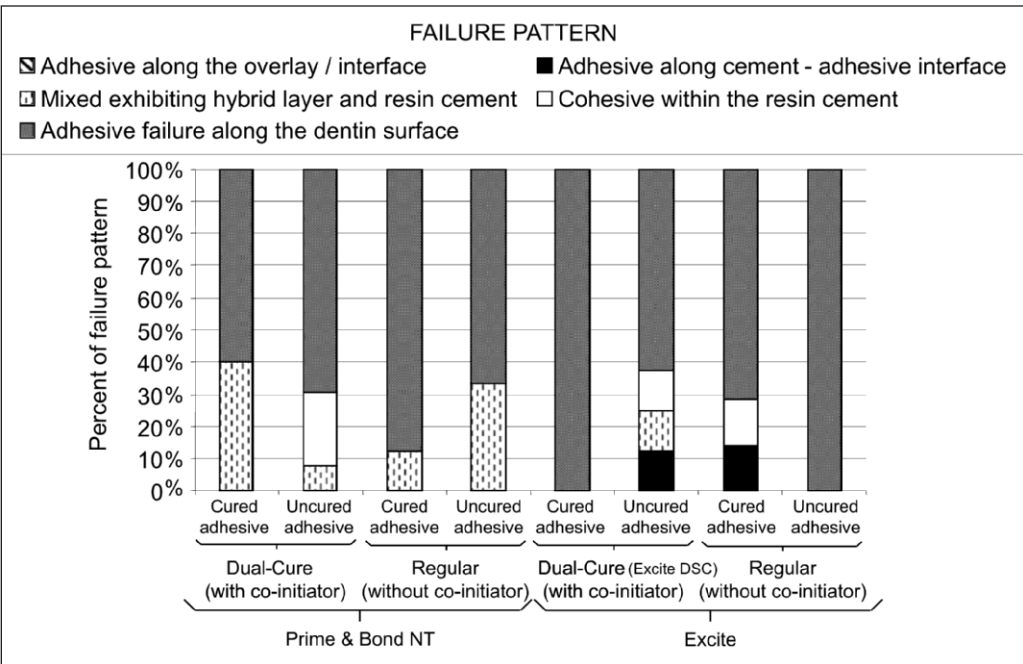


Figure 1. Proportional prevalence (%) of failure patterns for all experimental groups.

($p=0.0003$). The ANOVA analysis also detected a double interaction between “adhesive brand” and the “presence of co-initiator” factors. Therefore, the use of co-initiator resulted in lower bond strength for Prime & Bond NT, regardless of the curing mode of the adhesive layer, while no significant difference in bond strength was noted between teeth restored with Excite and those restored with Excite DSC.

Excite DSC exhibited higher bond strength than Prime & Bond NT Dual-Cure when the adhesive layer was either light-activated or left in the uncured state. However, when co-initiator was not added to the adhesive systems, Prime & Bond NT exhibited higher bond strength than Excite ($p=0.0052$).

Failure Pattern Analysis

Figure 1 shows the proportional prevalence (%) of the failure patterns in all experimental groups. All experimental groups had the highest percentage of failure mode classified as adhesive failure along the dentin surface.

DISCUSSION

The results of this study demonstrated that the effects of co-initiator on the bond strength of indirect restorations to dentin may depend on the product, rather than on the curing mode of the adhesive layer. The addition of co-initiator to Prime & Bond NT resulted in lower bond strengths than the use of the bonding agent without co-initiator, regardless of the curing mode of the adhesive layer. This finding may be related to the composition of the mixture between the bonding agent and

activator components. As both bonding agent and activator solution have high content of organic solvents, such as acetone and ethyl alcohol (Table 2), it is possible that such a mixture creates a solution with low content of monomers/activator components and a high concentration of organic solvents. According to Cho and Dickens,¹⁵ higher acetone concentration in such bonding solutions might, despite the drying step, allow residual solvent in the adhesive resin to remain. The residual content of organic solvent might have promoted the phase separation between hydrophobic and hydrophilic components and reduced free-radical poly-

merization initiated by both light exposure and aromatic sodium sulfinate salt, the self-polymerizing initiator. Thus, the resin polymerization of Prime & Bond NT Dual Cure may have been compromised and the mechanical properties of the adhesive resins impaired, as also observed by de Menezes and others.¹⁶ Therefore, the first research hypothesis of the current study was rejected for Prime & Bond NT. The effects of the higher amount of residual organic solvents might also explain why only the μ TBS promoted by Prime & Bond NT with co-initiator was lower than that of Excite and Excite DSC.

On the other hand, no significant difference in μ TBS was observed when Excite was used either with (Excite DSC) or without co-initiator, regardless of the curing mode of the adhesive layer. Therefore, the research hypothesis stating that the addition of co-initiator to the bonding agent provides higher bond strength regardless of the curing mode of the adhesive layer was invalidated for this adhesive system as well. These results are in agreement with those by Arrais and others¹⁴ regarding fifth-generation adhesive systems. The failure pattern predominantly located within the hybrid layer (Figure 1) demonstrates that possible chemical incompatibility between the resin cement and adhesive oxygen-inhibited layer¹⁷ at the top of the adhesive layer did not affect the bond strength of Excite when no co-initiator was used and the adhesive layer was light-activated prior to the resin cement application.

Lower bond strength was obtained when the adhesive layer was left in the uncured state compared to when

the adhesive layer was polymerized prior to resin cement application, regardless of the use of co-initiator. In contrast to this finding, another study demonstrated that the curing mode of the adhesive layer does not affect bond strength when light-activation is performed after placement of the indirect restoration for fifth-generation dual-cured adhesive systems.¹⁸ Such conflicting results might be related to the differences in method. While different from other studies evaluating the bond strength of indirect restorations,^{5,14,18-20} the current study attempted to simulate a clinical situation where final light-activation is delayed due to the time spent in removing resinous excess material from the margin of the indirect restoration. This delay may be considered long when a clinician is cementing only one indirect restoration, but it may correspond to the delay in light-activation when seating multiple indirect restorations or a metal-free ceramic bridge. For this reason, light activation of the indirect restoration was only performed five minutes after the indirect restoration was seated. Some authors have demonstrated that a significant portion of self-curing reaction takes place within this period just after placement of the indirect restoration.²¹⁻²³ Therefore, an increase in resin cement viscosity is expected, which decreases the reactive species mobility imposed by the initial formation of a highly cross-linked polymeric network.²⁴ The same change in viscosity promoted by the self-curing reaction would be expected in the combined layer composed of resin cement and uncured adhesive resin layer, so that monomer conversion of both the combined layer and adhesive resin infiltrated into demineralized dentin could be compromised. In addition, the higher content of acidic monomers from the uncured adhesive layer may have interfered with the amine present in the self-cure system, possibly resulting in less amine being available to participate in the light curing process with camphorquinone even when co-initiators were used. As a consequence, monomer conversion and the mechanical properties of resin cements may be compromised when the resin cements are allowed to self-cure during the five minutes prior to light-activation.

Within the light-activated groups, a difference in bond strength was observed in the groups restored with Prime & Bond NT and those restored with Prime & Bond NTD, while no significant difference was found between the Excite and Excite DSC groups. Considering the evidence that the failure pattern of all light-activated groups was predominantly located within the hybrid layer (Figure 1), which corresponded to the weakest region of the adhesive interfaces, and that the co-initiators have the same composition despite the presence of organic solvent in one of them, it is possible to speculate that both the presence and the presentation mode of co-initiators were the main reasons for the differences in bond strength for the light-activated

groups. In other words, the use of co-initiators presented as a solution resulted in lower μ TBS, while no significant difference in μ TBS was noted when a co-initiator presented as a salt was added to the bonding agent. Thus, the second research hypothesis, which stated that the presentation mode of the co-initiator (as a salt or diluted in organic solvent) is not related to the bond strength of indirect restorations regardless of the curing mode of the adhesive layer, was invalidated. Care must be taken during the adhesive application, and the vigorous air-drying step of dual-cured adhesive systems presenting co-initiators diluted in organic solvents should be performed to ensure that the most solvent content is properly evaporated before the next clinical steps.²⁵

The results obtained in the current study are based on *in vitro* conditions, which did not reproduce some variables observed with *in vivo* studies. Among all variables, the presence of pulpal pressure may be the one variable that deserves more concern regarding its effects on adhesive interface.²⁶⁻²⁷ As the mechanical properties and degradation of adhesive resin are related to the presence of water entrapped in the adhesive interface,²⁸⁻³⁰ pulpal pressure could lead to an increase in fluid content within the hybrid layer, therefore, the detrimental effects of water on the mechanical properties of adhesive resin within the hybrid layer and at the adhesive layers could be more pronounced.³¹⁻³² As higher solubility and water absorption are observed in some dual-polymerizing resin cements when they were allowed to self-cure,³³ the delayed light-activation of resin cement after indirect restoration placement could allow for a higher volume of water to infiltrate into the hybrid layer when the adhesive layer was left in the uncured state in a pulpal pressure simulation. Thus, the detrimental effects of water on the bond strength of indirect restorations could be more evident. However, only further studies can confirm this hypothesis.

CONCLUSIONS

Within the limitations of the current study, no difference in bond strength was observed when co-initiator was added to Excite, while the addition of co-initiator decreased the bond strength for Prime & Bond NT; therefore, the hypothesis stating that the presence of co-initiator would lead to higher bond strength was invalidated for the products evaluated in the current study. Such differences may be attributed to differences in the presentation mode of the co-initiator. Therefore, the second research hypothesis, which stated that the presentation mode of the co-initiator (as a salt or diluted in organic solvent) is not related to the bond strength of indirect restorations regardless of the curing mode of the adhesive layer, was rejected. The addition of organic solvent in the presentation mode of the

co-initiator decreased the bond strength of indirect restorations.

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