Original Article

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Influence of viscosity and polymerization mode on bond strength of dual-cure resin luting agent to dentin

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Abstract

Aim: To evaluate the influence of the viscosity and curing mode on the bond strength of two resin cements to dentin. **Methods:** Eight experimental groups were formed (n = 7) according to the dual-cure resin cements (Nexus 2 - Kerr Corp. and Variolink II - Ivoclar Vivadent), curing modes (dual-cure or self-cure) and viscosities (low and high). Resin cements were applied to pre-cured composite resin discs (2 mm thick, Sinfony -3M ESPE), which were fixed to bonded dentin surfaces. The restored teeth were either light-activated (XL3000 - 3M ESPE) or allowed to self curing only. After 24 h, the teeth were both mesiodistally and buccolingually sectioned to obtain bonded beam specimens (0.8 mm^2 cross-sectional area). Each specimen was tested in microtensile strength at a crosshead speed of 0.5 mm/min until failure. **Results:** Data (in MPa) were analyzed statistically by three-way ANOVA and Tukey's post-hoc test (pre-set á = 0.05). No significant difference was observed between resin cements (p=0.26) and viscosities (p=0.13), however, the curing mode affected the BS within the viscosities (p=0.01). Statistically significant difference was observed for low viscosity: Nexus 2: 23.8(10.6) (dual-cure) and 16.0(5.1) (self-cure); Variolink II: 28.7(8.7) (dual-cure) and 11.9(3.0) (self-cure). **Conclusions:** Light activation yielded higher bond strength for the low-viscosity versions of the resin cements.

Keywords: dentin bonding agents, resin cement, bond strength, dentin.

Introduction

The adhesive cementation techniques for indirect metal-free restorations use dual-cure resin luting agent, and the clinical success of these restorations depends on the quality of the restorative material and its bonding to the mineralized dental tissues, among other factors. The luting agents are a combination of dual-cure resin cement and a bonding agent, which is responsible by the adhesion between the tooth and the resin cement[1-5].

Resin cements can be dual-cure, only self-cure or only light-cure materials. Dual-cure resin cements are indicated in clinical situations when no light is available to polymerize the material and the self-curing component should compensate for the absence of light. In other situations, there is a loss of light because of either the distance between the light-curing tip and the luting agent or light attenuation through the thickness of the indirect restoration[6-10].

The resin cements present different viscosities, which can produce cement

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layers with different thicknesses. These differences on thickness may affect some properties of the material, such as strength, modulus of elasticity and rheology, which can interfere in restoration longevity. Resin cements provide a correct seating of the indirect restoration and are also responsible for the tooth-restoration interfacial sealing, which prevents the marginal leakage[11-14]. Viscosity variation for resin cements is obtained by modifying the proportion between monomer composition and filler particle content[15]. Nothing is known about the viscosities of resin cements and their influence when used with different polymerization modes (dual-cure or self-cure) on bond strength to dentin. Thus, the purpose of this study was to measure the bond strength of pre-polymerized composite discs to underlying tooth structure using dual-cure resin luting agents with two viscosities (high and low), which either were allowed to self curing in the absence of light or were exposed to light through the composite disc. In addition, the failure site morphology was analyzed and compared by material types and polymerization modes. The research hypothesis tested was that bond strength values would be significantly higher when the resin cement was subjected to light-activation (dual cure) than when they were only allowed to self curing. It was also hypothesized that the bond strength of low-viscosity dual-cure resin cement would be significantly lower than that of high-viscosity resin cement.

Material and methods

Specimen Preparation and Experimental Groups

The research protocol was approved by the Ethics Committee of the School of Dentistry of Piracicaba, University of Campinas, SP, Brazil (176/2006). Fifty-six freshly extracted, erupted, human third molars were used. The teeth were stored in a saturated thymol solution at 5 °C for no longer than 3 months[16]. They were then sectioned transversally in the middle third of the crown, using a diamond blade saw (Series 15HC Diamond, Buehler Ltd, Lake Bluff, IL, USA) on an automated sectioning device (Isomet 2000, Buehler Ltd) under water cooling, exposing areas of mid-crown dentin.

The exposed dentin surfaces were wet polished (APL-4, Arotec, Cotia, SP, Brazil) using 600-grit SiC paper in order to create a flat surface with standardized smear layer before application of the bonding agents. The prepared teeth were then randomly divided into 8 groups (n = 7).

Two dual-cure resin cements that present commercial versions in high and low viscosity, Nexus 2 (Kerr Corp., Orange, CA, USA) and Variolink II (Ivoclar Vivadent, Schaan, Liechtenstein), and their respective adhesive systems and silane primers (OptiBond Solo Plus and Silane Primer - Kerr Corp.; Excite DSC and Monobond S - Ivoclar Vivadent) were used (Table 1). Fifty-six pre-polymerized, light-cure composite resin discs, 2 mm thick and 10 mm in diameter (B2D shade, Sinfony, 3M ESPE, St. Paul, MN, USA), were prepared to simulate overlying laboratory-processed composite resin restorations. The surface of each disc was sandblasted with 50 µm aluminum oxide (Danville Engineering Inc, San Ramon, CA, USA) for 10 s (air pressure = 0.552 MPa; distance from the tip = 1.5 cm) and silanated with coupling agents according to manufacturers' instructions (Monobond-S or Silane Primer).

The adhesive systems and the resin cements were applied and used according to manufacturers' instructions. The resin cements were mixed previously in the proportion of 1:1 (catalyst and base paste) and were applied to the sandblasted surface of the pre-polymerized composite resin disc, which was placed on the dentin surface. The resin cements were light-cured for 40 s (XL 3000; 3M ESPE) through the

Table 1. Compositions of the resin cements used in this study.

Products (type)	Composition	Lot number
Variolink II(Resin cement)	Paste of dimethacrylates, Bis-GMA, TEGDMA, UDMA, inorganic fillers, ytterbium trifluoride, initiators, stabilizers, pigments, benzoyl peroxide.	High: J24363 Low: J19103 Base: J19730
Excite DSC(Adhesive system)	Adhesive resin: alcohol, phosphonic acid acrylate, HEMA, SiO_2, initiators and stabilizers, dimethacrylates.Activator: aromatic sodium sulfinate salt.	F54523
Nexus 2(Resincement)	Monomers of methacrylic acid esters, Ba–Al–borosilicate glass, chemical and photoinitiators.	High: 438681 Low: 452344 Base: 452365
OptiBond Solo Plus(Adhesive system)	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.	Adhesive: 487047 Activator: 100914
Monobond S(Silane coupling agent)	Ethanol, 3- methacryloxy-propyl-trimetoxy-silane	H24764
Silane Primer(Silane coupling agent)	Ethylalcohol, organosilaneester.	32667

Abbreviations: TEGDMA: Triethylene glycol dimethacrylate; Bis-GMA, bisphenol A diglycidyl ether methacrylate; UDMA, urethane dimethacrylate; HEMA: 2-hydroxyethyl methacrylate; GPDM: glycerol phosphate dimethacrylate.

composite resin disc or were allowed to self curing only with a load of 0.5 kg applied horizontally for 5 min. In order to facilitate specimen gripping during bond testing, a 3-mm-thick block of self-cure composite resin (Concise, 3M of Brazil, Sumaré, SP, Brazil) was added to the untreated, pre-polymerized composite resin surface.

Microtensile bond strength

Restored teeth were stored in distilled and deionized water at 37° C for 24 h and were then vertically serially sectioned into several 1.0-mm-thick slabs. Each slab was further sectioned perpendicularly to produce bonded beam specimens with 0,8mm^2 in cross-section. Each bonded beam was attached to the grips of a microtensile testing device with cyanoacrylate glue (Super Bonder; Henkel/Loctite, Diadema, SP, Brazil) and tensioned in an universal testing machine (4411; Instron Co., Canton, MA, USA) at a crosshead speed of 0.5 mm/min until failure. After testing, the specimens were carefully removed and the cross-sectional area at the site of fracture was measured to the nearest 0.01 mm with a digital caliper (mod. 727-6/150, Starret Ind. e Com. Ltda., Itu, SP, Brazil). The specimen cross-sectional area was divided by the peak tensile load at failure to calculate stress at fracture (in MPa). A single failure stress value was then calculated for each tooth by averaging the values of 5 tested beams from that tooth.

A three-way ANOVA (two resin cements, two polymerization modes and two viscosities) was performed to determine the effect of these major factors on tensile strength. Tukey's post-hoc test was used to detect pair-wise differences among the experimental groups. All statistical testing was performed at a preset α of 0.05.

Failure pattern analysis

Fractured surfaces of tested specimens were sputter coated with gold (MED 010, Balzers, Balzer, Liechtenstein) and examined by a single individual using a scanning electron microscope (VP 435, Leo, Cambridge, England). Failure patterns were classified as: (1) adhesive failure between adhesive resin and dentin; (2) cohesive within the adhesive resin, (3) adhesive failure between adhesive and resin cement (4) cohesive within the resin cement, (5) mixed failure involving different structures of dentin-resin disc bonded interface. Representative areas of the failure patterns were photographed (85× to 1,900×).

Results

Summary statistics for the different experimental groups are shown in Table 2. Three-way ANOVA indicated that the curing mode factor significantly influenced tensile strength results ($p = 0.0005$). The statistical analysis revealed difference only for curing mode ($p= 0.0004$), no significant differences for the triple interaction (resin cement × curing mode × viscosity, $p = 0.4303$) or for interaction between cement × viscosity ($p = 0.1710$) and cement × curing mode ($p = 0.1312$). The analysis only indicated an interaction between the viscosity x curing mode ($p = 0.0127$).

Table 2. Means (standard deviation) of bond strength to dentin for Nexus 2 and Variolink II resin cements as a function of viscosity and curing mode (in MPa).

Resin cement	Viscosity	Dual-cure	Self cure
Nexus 2	Low	23.8 (10.6) Aa	16.0 (5.1) Ab
Nexus 2	High	20.2 (4.7) Aa	18.9 (6.5) Aa
Variolink II	Low	28.7 (8.7) Aa	11.9 (3.0) Ab
Variolink II	High	16.5 (4.7) Aa	13.1 (6.4) Aa

Similar letters indicate no statistically significant difference among values (uppercase letters compare viscosities within the same resin cement and lowercase letters compare curing modes).

When looking at data with respect to differences in curing mode, the bond strength of the low viscosity version of the resin cements was affected by curing mode ($p< 0.05$), while light-activation did not increase the bond strength of high viscosity versions ($p> 0.05$). The type of resin cement and the viscosity did not affect the bond strength ($p> 0.05$).

Figure 1 shows the proportional prevalence (%) of the failure patterns in all experimental groups, and representative images depicting failure modes are presented in Figures 2 and 3. Adhesive failures along the dentin surface were observed for all groups, except for Nexus 2 in low-viscosity and self cure mode. For Variolink II in high viscosity and dual-cure mode, half of the specimens had adhesive failures (type 1) (Figures 2A and 2B). High- or low-viscosity dual-cure Nexus 2 exhibited high incidence of cohesive failures within the adhesive resin and adhesive failures between adhesive and resin cement (Figures 3A and 3B, respectively). Except for high-viscosity dual-cure Nexus 2, cohesive failures within the resin cement was observed for all groups; however, it occurred mainly for self-cure groups (Figure 3C). Mixed failures involved two or more types of failures (adhesive and cohesive failures) in the same fractured end of specimens and all groups with Variolink II showed mixed failures (Figures 2C, 2D, and 2E).

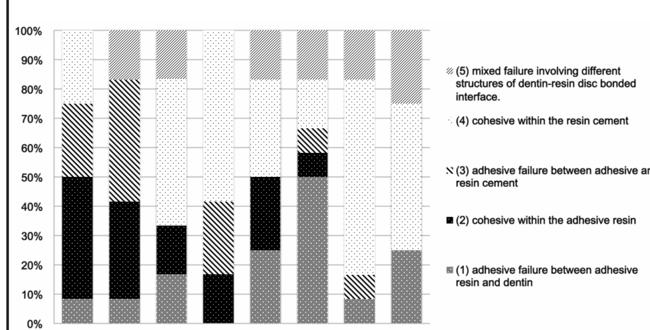

Fig. 1. Distribution (%) of failure modes among experimental groups.

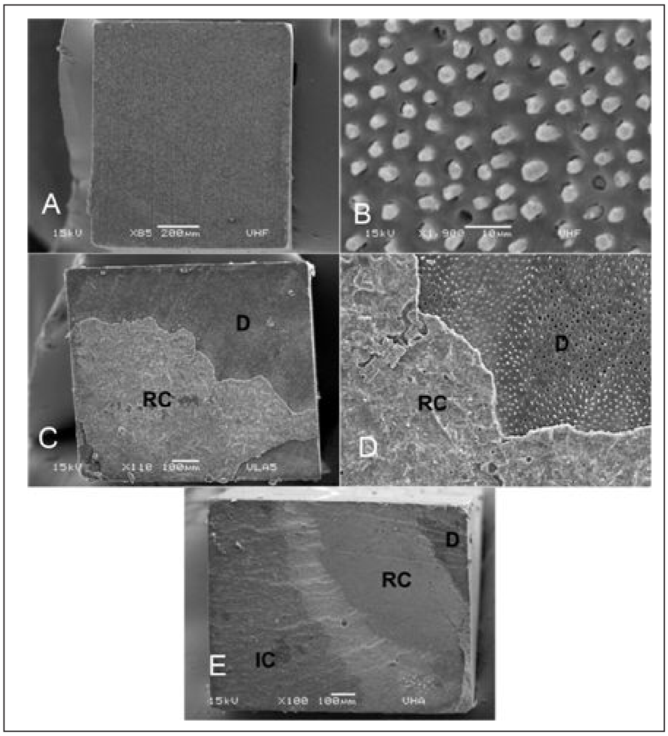

Fig. 2. (A) Fractured end of specimen cemented with Variolink II (high viscosity and dual-cure) exhibiting adhesive failure along dentin surface (original magnification X85). **(B)** Higher magnification of Figure 2A revealing fractures located predominantly within the hybrid layer and resin tags inside the dentinal tubules (original magnification X1900). **(C)** Fractured end of specimen cemented with Variolink II (low viscosity and self-cure) exhibiting mixed failure mode, characterized by adhesive and cohesive failures within the resin cement (original magnification X100). D- dentin; RC- resin cement. **(D)** Higher magnification (X500) of Figure 2C demonstrating RC (resin cement) and D (dentin) within same fractured surface. **(E)** Fractured end of specimen cemented with Variolink II (high viscosity and self-cure) exhibiting mixed failure mode, characterized by adhesive and cohesive failures within the resin cement and within indirect composite (original magnification X100). D- dentin; RC- resin cement; ID- indirect composite.

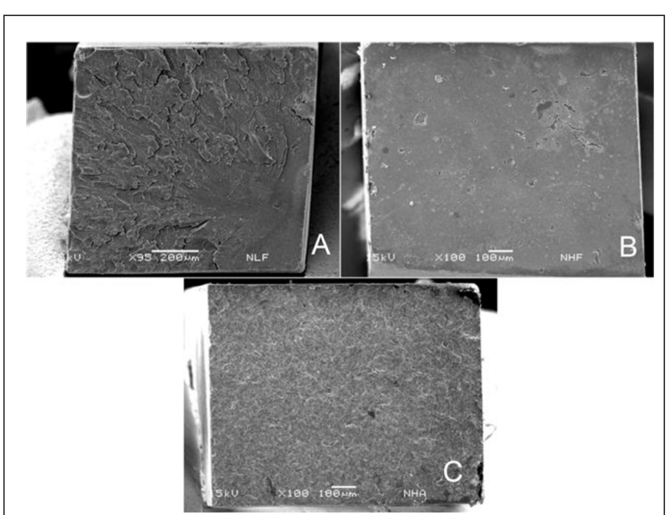

Fig. 3. (A) Fractured end of specimen cemented with Nexus 2 (high viscosity and dual-cure) exhibiting cohesive failure within the adhesive resin (original magnification X95). **(B)** Fractured end of specimen cemented with Nexus 2 (high viscosity and dual-cure) exhibiting adhesive failure between adhesive and resin cement (original magnification X100). **(C)** Fractured end of specimen cemented with Nexus 2 (high viscosity and self-cure) exhibiting cohesive failure within the resin cement (original magnification X100).

Discussion

A more effective bonding to dentin can increase the strength of remaining dental structures and reduce microleakage between the tooth and restoration, which are important to tooth longevity[17,18]. For supporting use of these materials, we tested the research hypothesis that bond strength values would be significantly higher when the resin cements were light-activated, which was confirmed only for low-viscosity versions. The hypotheses that the bond strength of resin cements in low-viscosity versions would be significantly lower than those in high-viscosity cements was rejected, since no statistical difference was noted between low- and high-viscosity materials. The low and high versions of resin cements are indicated for different purposes[15,19], but the compositions that form both viscosities did not influence the bond strength. Since the resin cements reach high monomer conversion after light curing, only the low-viscosity resin cements were able to produce higher bond strength.

The high-viscosity version of both resin cements showed no significant difference on bond strength irrespectively of being light activated or not, since the increased amount of fillers improves the mechanical properties of the cement. The bond between the monomer components and filler particles by treatment of these particles, such as silanization, and the higher amount of fillers promote the increase of the cohesive strength of the resin cement[19]. However, the low-viscosity formulation of the resin cements did not maintain the bond strength mean value when the materials were allowed to self curing only. Light activation of low-viscosity resin cements produced higher bond strength to dentin than self curing alone[20]. Cohesive failures within the resin cement occurred predominantly in those specimens in which the resin cements were allowed to self curing only (Figure 1).

Since the self curing reaction alone does not reach the monomer conversion promoted by light[6,10], the resin cement layer may be the weak point at the resin-dentin interface. Thus, in the present in vitro study, when the resin-dentin interface was under tension, the resin cement layer tended to fracture more easily than other parts of this interface. Depending on the resin cement and its viscosity, light-curing caused different types of failure (Figures 1 to 3). For the self-cure resin cements, the predominant failure pattern was the cohesive failure in resin cement, indicating that the lack of light activation reduced the cohesive strength of the cement (Figures 2C, 2D, 2E and 3C). The dual-cure resin cements presented different failure patterns, depending on the type of adhesive systems used (Figures 2A, 2B, 3A and 3B).

The luting agents tested in this study showed no significant difference when compared by different viscosities and curing modes. These materials showed the same results in terms of bond strength to dentin, corroborating the findings of Hikita et al.[21] (2007). Other studies showed that Variolink II presented higher bond strength than Nexus 2, but they did not report viscosity of these resin cements used[4,22]. Regarding the curing mode, few resin cement systems (Nexus 2, Kerr Co.; Variolink II, Ivoclar Vivadent; Panavia F, Kuraray; RelyX

ARC and RelyX Unicem, 3M ESPE) seem able to produce proper degree of conversion and high bond strength to dentin[6,13-15,17].

The bonding agents (OptiBond Solo Plus and Excite DSC) used with resin cements are etch-and-rinse, two-step systems. They are simplified single-bottle adhesive systems that present a low pH value and might jeopardize resin cement conversion reducing the bond strength, if only autopolimerized[23]. As adhesive systems are spread into a thin layer, an incompletely polymerized resin monomer layer is formed on the adhesive surface. The oxygen inhibition layer formed by uncured acidic simplified etch-and-rinse systems impairs the adhesion between bonding agents and chemical-, light- or dual-cured resin-based restorative materials. The adverse reaction involves the uncured acidic adhesive layer and the tertiary amine catalytic component of the resin cement[23,24].

Both adhesives contain an aromatic sodium sulfinate salt as a co-initiator to develop dual-cure reaction. Excite DSC uses the co-initiator impregnated in the microbrush tip as a white salt powder, while OptiBond Solo Plus has an activator bottle that contains the benzene sulfinic acid sodium salt in an alcoholic solution[4,20]. In this study, the addition of these co-initiators to the bonding agents contributed to preserve the bond strength of the high-viscosity cements without light exposure (self curing), since the adhesive was not polymerized and the co-initiator could mix with cement, increasing the polymerization of cement. The same, however, did not occur with the low-viscosity version because the mixture of liquid solutions from the adhesive and activator with a hydrophobic flowable resin cement material may be poor and a larger amount of monomers requires a higher formation of free radicals for an efficient polymerization[17]. These mixtures involve an aqueous solution containing sodium sulfinate salt and a hydrophobic material, which resulted in lesser polymerization and lower bond strength for the self-cure cements.

It may be concluded that light activation yielded higher bond strength for the low-viscosity versions of the resin cements.

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