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Structural reliability and bonding performance of resin luting agents to dentin and enamel

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ABSTRACT

This study evaluated the bonding performance and structural reliability of indirect resin composite restorations luted to dentin and enamel. Four resin luting agents were tested in the study, namely three total-etch materials (Allcem; Allcem Core; and RelyX ARC) and one self-adhesive material (RelyX U200). The materials were characterized with degree of conversion (DC) and pH analyses, and used to lute resin composite restorations to dentin or enamel (n = 6). The restorations were prepared for microtensile bond strength (μTBS) testing (DL500). Data were analyzed with ANOVA or Kruskal-Wallis and Tukey/SNK (α = 0.05). Weibull analysis was used to verify the structural reliability and characteristic strength of dental bonds. While RelyX ARC exhibited greater DC, RelyX U200 was the most acidic material of the study. Allcem and RelyX U200 demonstrated greater and lower μTBS, respectively, at both dentin and enamel. Comparing μTBS of dentin and enamel, RelyX ARC and RelyX U200 produced higher bonds in dentin. RelyX U200 showed ~100% of adhesive failures, differing from other groups. Structural reliability was higher for Allcem Core in dentin and for RelyX U200 in enamel; the characteristic strength was higher for Allcem and lower for RelyX U200. Conventional resin luting agents performed better than the self-adhesive material, at both dentin and enamel. Among the conventional materials, Allcem demonstrated an overall greater bonding ability and lower probability to failure. DC and pH of resin luting agents did not influence on their bonding performance.

1. Introduction

Dental bonding of indirect restorative materials or intracanal post systems using resin luting agents can be achieved by following two distinct adhesive strategies: conventional/total-etch or self-adhesive [1]. While the former require the application of an acidic substance followed by adhesive agents prior to the bonding procedure in order to allow proper demineralization and resin infiltration to the substrate; the latter eliminates the pre-use of any adhesive system, since the luting agent itself possesses the ability to demineralize and infiltrate dentin and enamel with resin monomers [2]. Despite their adequate clinical applicability, the current literature still diverges on which adhesion strategy would perform better when bonding indirect composite restorations [3,4], so that new studies on this topic are still necessary, attempting to compare the bonding performance of different materials to both dentin and enamel.

Bond strength testing has been broadly considered for verifying the adhesion/retention of restorative materials to the tooth. However, these tests usually lack in standardization, leading to great variation data [5–7]. In light of improving the reliability and interpretation of bond strength tests, studies have been applying Weibull statistics as an additional measure of strength data, offering a strong theoretical basis of understanding the structural reliability and strength properties of fractured samples, especially in dental applications [8]. To date, there are only few studies focusing on the evaluation of the structural reliability of dental bonds obtained with the application of resin luting agents.

Hence, the present study aimed to evaluate the bonding performance and structural reliability of resin composite indirect restorations luted to dentin or enamel using distinct resin luting agents. The hypothesis of the study was that conventional resin luting agents would present higher bonding performance than self-adhesive materials, regardless of the dental substrate.

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2. Materials and methods

This study evaluated two variable factors: dental substrate (dentin or enamel) and resin luting agent (total-etch or self-adhesive). Four materials were investigated: three total-etch (Allcem; Allcem Core; and RelyX ARC) and one self-adhesive (RelyX U200). Information on their manufacturer, chemical composition, inorganic filler fraction, and bonding protocol is given in Table 1. All materials were applied following the manufacturer's instructions.

2.1. Degree of conversion

For degree of conversion (DC) analysis (n = 3), each material was measured (~5 μL; 5 mm diameter × 0.5 mm thickness) using Fourier transform mid-infrared spectroscopy (Shimadzu) in ATR mode, at a range of 1750 to 1550 cm^{-1}, resolution of 8 cm^{-1}, and mirror speed of 2.8 mm/s [9]. After baseline reading (non-polymerized), photo-activation was performed for 40 s using a light emitting diode (LED) curing unit (Radii; SDI) with irradiance of 1000 mW/cm^2 (measured using an optical power meter; Ophir Optronics), and a new measurement was obtained (polymerized material). The absorbance bands at 1637 cm^{-1} (methacrylate group, C=C) and 1607 cm^{-1} (ester group, C=O) were used to calculate the DC (%) as follows [10]:

$$DC = 1 - \frac{\text{Cured (area under 1637/ area under 1607)}}{\text{Uncured (area under 1637/ area under 1607)}} \times 100$$

2.2. pH analysis

For pH analysis, ~3 mL of each material was placed into a vial and tested with an electrode of a digital pHmeter (model AN2000, Analion) after proper calibration steps using buffer solutions of pH 7.0 (neutral buffer) and 4.0 (acidic buffer). All pH readings were performed in triplicate (n = 3) and at constant temperature of 25 °C.

Table 1
Resin luting agents tested in the study and their information on manufacturer, lot number, and cementation protocol.

Resin cements (Manufacturer)	Chemical composition	Filler fraction	Cementation protocol in dentin/enamel
Allcem (FGM) Allcem Core (FGM)	Organic: Bis-GMA, Bis-EMA, TEGDMA, photoinitiators, stabilizers Inorganic: Ba–Al–Si glass microfillers, SiO_2 nanofillers, pigments	66-67 wt% 62 wt%	Perform the following clinical steps: "a" (15 s in dentin and 30 s in enamel), "b", "c" (10 s), "d" (10 s), "e" (10 s), "f" (20 s), and "g"
RelyX ARC (3 M ESPE)	Organic: Bis-GMA, TEGDMA, photoinitiators Inorganic: zirconia/silica fillers, pigments	67.5 wt %	Perform the following clinical steps: "a" (15 s in dentin and 30 s in enamel), "b", "c" (10 s), "h" (20 s, only in dentin), "c" (10 s), "i" (10 s), "f", and "g"
RelyX U200 (3 M ESPE)	Organic: acidic monomers, TEGDMA, acids, dimethacrylates, photoinitiators Inorganic: glass fillers, SiO2, pigments, sodium persulfate, glass fibers,	~70 wt %	Perform the clinical step "g".

Clinical steps: "a" – acid-etching with a 37% phosphoric acid gel; "b" – water rinsing for removal of the acidic gel; "c" – air-drying with an oil-free air spray; "d" – application of the Ambar (FGM) bonding agent; "e" – solvent evaporation (10 s); "f" – light-activation (20 s); "g" – application of the resin cement; "h" – application of the primer solution of the Scotchbond Multipurpose (3 M ESPE) adhesive system; and "i" – application of the bonding agent of the Scotchbond Multipurpose (3 M ESPE) adhesive system.

2.3. Microtensile bond strength

Forty-eight bovine incisors were cleaned, stored in 1.23% chlorhexidine solution (2 h), and immersed in distilled water (4 °C) until use. The root was removed and the crowns were randomly allocated into two groups: dentin or enamel. The crowns of the dentin group were finished with #600-grit SiC abrasive paper until mid-dentin exposure; crowns of the enamel group were finished with #600-, #800- and #1200-grit SiC papers for 1 min each. Forty-eight resin blocks (Opallis; FGM) were prepared (5 × 5 × 5 mm) following an incremental technique, which had their upper face finished with #600-, #800- and #1200-grit SiC papers for 1 min each, followed by acid-etching with 37% phosphoric acid gel (Condac 37; FGM) for 15 s, washing with distilled water, and treatment using silane (Prosil; FGM) for 20 s. Dental substrates were treated as demonstrated in Table 1. Each luting agent was mixed and immediately applied onto the treated face of the resin blocks and on the treated dentin/enamel samples (n = 6). A load of 500 g was applied over the luted blocks for 6 min, followed by photo-activation during 40 s at each interface.

After water storage (24 h at 37 °C), the samples were sectioned at two perpendicular directions using a precision cutting machine (Isomet 1000; Buheler), resulting in specimens of ~0.8 mm^2 of cross-sectional area. The specimens were stored in distilled water for 24 h (37 °C), fixed to a jig dispositive with cyanoacrylate adhesive (Super Bonder Gel, Loctite), and tested in a universal testing machine (DL200, EMIC) for microtensile bond strength (μTBS) test at a crosshead speed of 1 mm/min. The fractured specimens were carefully observed under a stereomicroscopic loupe (40 × mag), and failure modes were classified as adhesive, cohesive in resin composite, cohesive in dentin/enamel, or mixed failure. Two specimens of each group were evaluated using SEM analysis (VEGA 3, TESCAN Brno).

2.4. Statistical analysis

Data were analyzed with Sigmaplot version 12 (Systat Software Inc.). The data from DC and pH had a normal distribution (Kolmogorov-Smirnov test) and were analyzed using 1-way Analysis of Variance (ANOVA) followed by Tukey post-hoc test ($p < 0.05$); whereas the data from μTBS did not have a normal distribution, being analyzed using ANOVA on ranks and Student-Newman-Keuls ($p < 0.05$). The experimental unit considered for the statistical analysis of bond strength data was the μTBS specimen, so the average of specimens was calculated and followed the mean group. Reliability and probability of failure were analyzed by Weibull analysis and 95% confidence intervals [11].

3. Results

DC was higher ($p \leq 0.007$) for RelyX ARC (78.4% ± 1.9) than Allcem Core (64.9% ± 1.7), Allcem (56.6% ± 3.3), and RelyX U200 (56.4% ± 5.6), which have not differed between each other ($p \geq 0.067$). Concerning pH, RelyX U200 showed the lowest value (2.2 ± 0.1), which was lower than the other materials ($p < 0.001$). Allcem Core (4.9 ± 0.3) and RelyX ARC (4.8 ± 0.4) showed similar pH values ($p = 0.880$) and higher than Allcem (3.8 ± 0.1) ($p \leq 0.003$).

Fig. 1A shows the bond strength results of the study. The highest resin-dentin bonds were obtained with the application of Allcem (31.9 MPa ± 8.5), followed by RelyX ARC (28.8 MPa ± 10.8), Allcem Core (25.6 MPa ± 5.1), and RelyX U200 (13.9 MPa ± 3.2). All groups differed statistically from each other ($p \leq 0.031$). The resin-enamel bonds were also higher for Allcem (30.2 MPa ± 6.0), followed by Allcem Core (25.5 MPa ± 7.3), RelyX ARC (21.0 MPa ± 5.6), and RelyX U200 (10.5 MPa ± 1.9); all groups were different from each other ($p \leq 0.002$). Comparing the μTBS on dentin and enamel, reduced bond strength values were observed in enamel only upon the use of RelyX ARC and RelyX U200 ($p \leq 0.021$). Adhesive failures were more frequent (>70%) for all groups at both dental substrates; whereas cohesive failures in enamel were

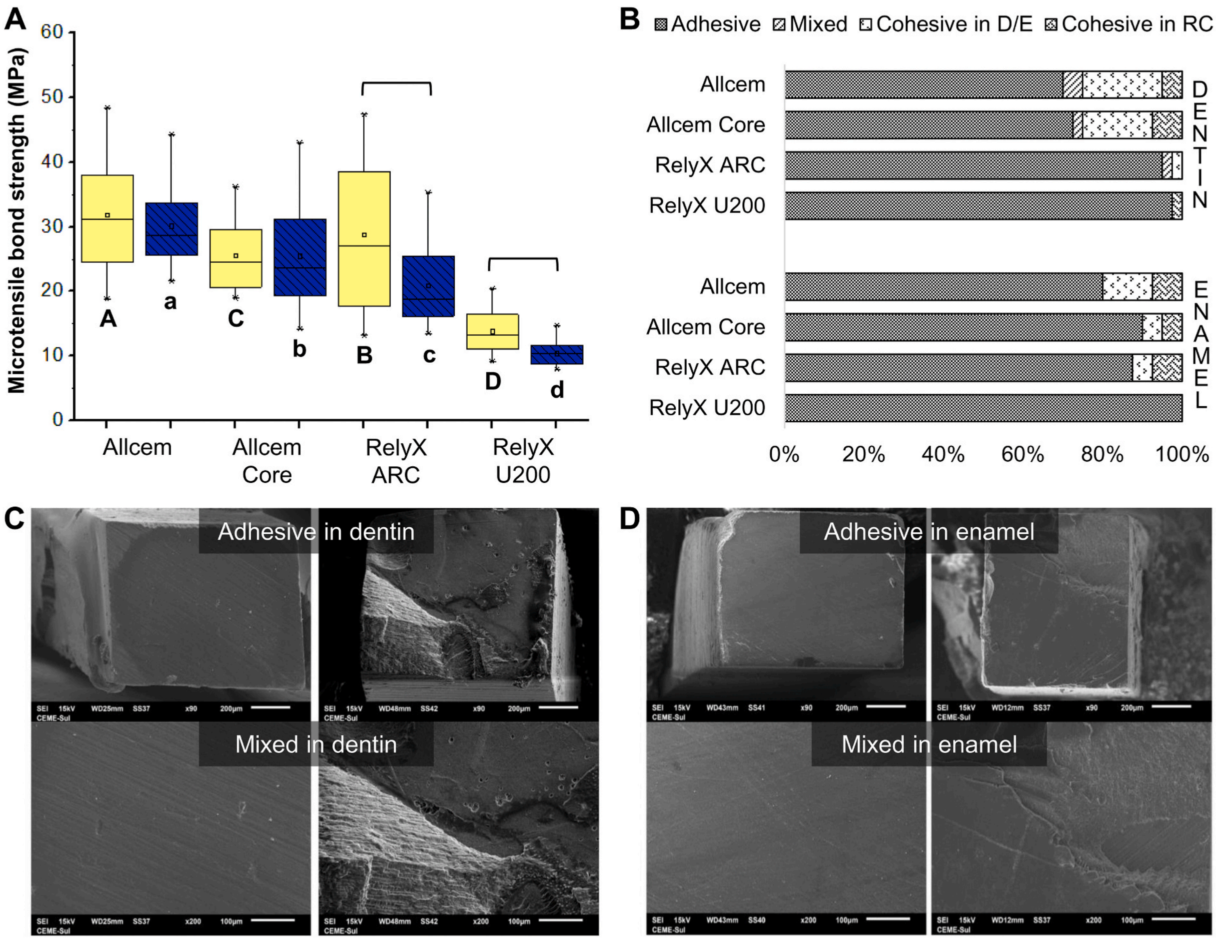

Fig. 1. (A) Box-plot graph showing the microtensile bond strength results obtained in this study. Distinct uppercase letters in non-dashed yellow box-plots indicate statistical differences among groups bonded to dentin, whereas distinct lowercase letters in dashed blue box-plots represent statistical differences among groups bonded to enamel ($p < 0.05$). Box-plots connected by a line indicate a statistical significant difference between dentin and enamel within the same group ($p < 0.05$). (B) Distribution of the failure modes of groups tested. Representative SEM micrographs showing two failure patterns occurred on dentin (C) and enamel (D). Upper and lower images are given at 90 × and 200 × magnification, respectively. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

observed for groups luted with Allcem and Allcem Core (Fig. 1B). Fig. 1C–D shows representative SEM micrographs of adhesive and mixed failures at dentin and enamel.

Table 2 shows the results for the Weibull analysis on dentin and enamel. In dentin, structural reliability was greater for Allcem Core than RelyX ARC, whereas in enamel RelyX U200 showed a more reliable adhesive interface than Allcem Core. Concerning the characteristic strength, RelyX U200 exhibited the weakest behavior at both substrates; Allcem showed the strongest characteristic, which was greater than Allcem Core in dentin and RelyX ARC in enamel. The correlation coefficient was greater than 0.83 for all analyses.

Table 2
Weibull modulus (*m*), characteristic strength (σ_0, in MPa), and correlation coefficient (r^2) for the groups tested in the study at dentin and enamel substrates.

Groups	Dentin			Enamel		
	m (95% CI)	σ_0 (95% CI)	r^2	*m* (95% CI)	σ_0 (95% CI)	r^2
Allcem	4.4 (3.6–5.4)^{AB}	36.1 (34–39)^{A}	0.92	6.1 (5.0–7.6)^{AB}	32.0 (30–34)^{A}	0.89
Allcem Core	5.8 (4.8–7.3)^{A}	26.3 (25–28)^{B}	0.83	4.2 (3.5–5.2)^{B}	28.9 (27–32)^{A}	0.92
RelyX ARC	3.0 (2.5–3.7)^{B}	32.6 (30–37)^{A}	0.91	4.5 (3.7–5.6)^{AB}	22.6 (22–25)^{B}	0.88
RelyX U200	5.2 (4.3–6.5)^{AB}	15.3 (15–16)^{C}	0.91	6.7 (5.5–8.3)^{A}	10.8 (11–11)^{C}	0.87

Distinct letters indicate statistically significant differences among the groups ($p < 0.05$).

4. Discussion

In this study, three conventional (total-etch) luting agents were tested (Allcem, Allcem Core, and RelyX ARC), opposed by only one self-adhesive material (RelyX U200). It has been hypothesized that the former would present greater bonding performance than the latter, which was indeed accepted. While RelyX U200 presented nearly 46–57% less adhesion ability to dentin as compared to conventional materials, the reduction of adhesiveness was even greater in enamel (47–64%). One may consider that the pre-etching step (total-etch protocol) has probably created a more adequate micro-retentive scenario for the bonding of indirect composite restorations [12]. Here, the more acidic behavior of RelyX U200 as compared to the other luting agents was not sufficient for the effective demineralization of dental mineral phase and consequent resin infiltration, which are paramount for the formation of adequate hybrid layers. While total-etch luting agents produce a 4-5 μm-thick hybrid layer, self-adhesive materials tend to create a less thick interface (2–3 μm or less) [13]. Of note, the adhesive interface produced with the application of self-adhesive luting agents is currently gaining a new terminology, namely “nanohybrid layer” or “interdiffusion zone” [14]. From the present bond strength data shown in Fig. 1A, RelyX U200 demonstrated the least bonding performance of the study, probably due to the formation of a non-uniform and weak nanohybrid layer [1]. This corroborates the ~100% adhesive failures observed for RelyX U200 groups. Overall, the greater bond strength results and the occurrence of mixed and cohesive failures in the other groups may indicate that total-etch luting agents may yield the best option for bonding indirect restorations to both dentin and enamel.

It is noteworthy that the adhesive strategy of the luting agent does not seem to be the only influencing variable, since differences in bond strength were also identified among the restorations luted with different total-etch materials. One aspect that may explain this result is the inherent composition of each luting agent [2]. While Allcem and Allcem Core share the same ingredients (Table 1), differing only with regard to their filler content (4–5% higher in the former); Allcem and RelyX ARC share a similar amount of fillers, but a different organic content. It seems that both the presence of a more heterogeneous monomer composition and a high filler loading level have contributed for the better bonding ability of Allcem. However, one could have expected that RelyX ARC would demonstrate the best adhesion ability due to its high degree of conversion [15], which was 12% and 21% higher than Allcem Core and Allcem, respectively. Here, it is of utmost importance to understand that the greater the polymerization level of methacrylate-based resin materials, the greater their volumetric shrinkage and development of polymerization stress [16]. It is reasonable to assume that the amount of polymerization stress originated during bonding of the indirect restorations with RelyX ARC was considerably higher than that obtained with the use of the other resin luting agents, resulting in the formation of less uniform resin-dentin/resin-enamel interfaces. In contrast, dental hybridization was probably more uniform with the application of Allcem, resulting in greater μTBS values at both dentin and enamel. Overall, it seems that for methacrylate-based formulations, the bonding ability of resin luting agents may be more negatively impaired by the expenses of polymerization stress rather than due to a reduced polymerization level of the material, although this deserves further investigation.

Two luting agents showed better bonding results in dentin than in enamel: RelyX ARC and RelyX U200. Enamel is a highly mineralized substrate, benefiting from the use of total-etch materials [3]. It may be considered that RelyX U200 offers insufficient acidity, impairing the effective and selective removal of minerals from enamel and the acquisition of adequate mechanical interlocking between luting agent and tooth [17]. One may also suggest that the acidity of RelyX U200 was neutralized once the material entered in contact with dentin/enamel, since calcium and phosphate minerals found at both dental substrates could have buffered the H^+ ions formed during the application of the self-adhesive material [18]. This effect could be importantly investigated in further studies upon the monitoring of pH changes (kinetic study [19]) once the luting agent gets in direct contact with dentin and enamel. Surprisingly, RelyX ARC has also showed less bonding ability in enamel than in dentin, so that other factors rather than solely the adhesive strategy should be considered to explain this result. For instance, enamel is stiffer than dentin, which may have increased the negative effects of polymerization stress within the adhesive interfaces [20], as discussed earlier. Of note, RelyX ARC resulted in high conversion of monomers, so that volumetric shrinkage and polymerization stress were probably high. No less important, the hydrophilic composition of RelyX ARC [21] might have reduced its wetting ability in hydrophobic enamel [22]. Indeed, discrepancies in the polarity of luting agent and substrate may impair the formation of an adequate adhesive interface [23], explaining the ~28% less bonding ability of RelyX ARC in enamel as compared to dentin.

This study investigated the effects of different luting agents on the structural reliability and characteristic strength of dental bonds. Allcem Core demonstrated a dual result, displaying the greatest and the lowest Weibull modulus in dentin and enamel, respectively, thus suggesting that the substrate plays a role in the reliability of dental bonds. Dentin is a more compliant substrate than enamel, presenting flexible properties that guarantee some deformation under loading circumstances; on the other hand, enamel is brittle, showing a low compliance behavior [22]. Considering that Allcem Core contains the least amount of fillers of the study (62 wt%), and consequently the most compliant behavior, it would be reasonable to assume that this luting agent would contribute for the creation of the most reliable bonds in dentin, but not in enamel. Likewise, RelyX U200 is the less compliant material tested, since it is comprised of ~70 wt% of fillers, contributing for its better Weibull modulus in enamel [24]. Once again, it seems that structural reliability tends to increase as soon as the compliance of luting agent and substrate matches to each other.

An interesting finding of this study shows that the best reliability was not verified upon the highest bonding performance. Notably, a reliable structure represents the lower likelihood to fracture occur at low level of stress [8], and Weibull modulus is a statistical parameter that relates to a narrower variation of data [11]. This may explain the reliable resin-enamel bonds obtained with RelyX U200, despite the lower bonding ability. Concerning the characteristic strength, it seems that this measure followed a similar tendency as verified with the bond strength results. Indeed, Allcem exhibited the strongest behavior, opposed by RelyX U200 with the weakest performance, suggesting that the characteristic strength is a mechanical-related measure of failure prediction [8]. Thus, we may consider that Allcem was the luting agent with the best overall reliability, showing the lowest probability of failure, regardless of the dental substrate tested, becoming a good option of resin luting agent for bonding resin composite indirect restorations.

Last but not least, one may consider that the use of bovine teeth and not human samples of enamel and dentin for conducting the present study could have diminished the extrapolation of our findings to the clinical level, but some studies have already demonstrated that bovine teeth have closeness of enamel microstructure to human enamel [25] and similar dentinal morphology to human dentin [26]. Also, according to a systematic review and meta-analysis study [27], bovine teeth can be a reliable substitute for human ones on bond strength studies of adhesive systems to both enamel and dentin substrates, so that the same trend could be expected when considering dental resin luting agents as the bonding material, as investigated in our study. Therefore, we may assume that the present findings are relevant to the scientific community, helping the dental practitioner during the bonding process of indirect resin composite restorations with resin luting agents.

5. Conclusions

In this *in vitro* study, total-etch resin luting agents performed better than the self-adhesive material, at both dentin and enamel, with Allcem resulting in an overall greater bonding ability and lower probability to failure. Degree of conversion and pH of the luting agent did not seem to influence on the bonding performance of resin luting agents.

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