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RESEARCH ARTICLE

Bond Strength to Dentine and Degree of Conversion of Adhesive Systems

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ABSTRACT

Objectives: **The study evaluated the bond strength, degree of conversion and fracture pattern of the hybrid layer in cement-dentin adhesion techniques.**

Materials and Methods: **Bovine teeth dentin blocks were divided according to the cementation techniques: G1- Conventional three-step adhesive and dual-curing resin cement; G2- One-step self-etch adhesive and dual-curing resin cement; G3-Self-adhesive resin cement; and G4- Self-adhesive primer and dual-curing resin cement. Specimens were submitted to thermal cycling, chemical aging or water storage. Bond strength was evaluated by testing machine; fracture pattern observed by optical microscopy, and degree of conversion evaluated in micro-Raman spectrophotometer. Data were analyzed by two-way ANOVA and Tukey's test (α=0.05).**

Results: **G1 showed greatest bond strength ($p<0.001$) followed by the G2 with higher values compared to G3 and G4, and no difference between them. For G2, G3 and G4, the thermal cycling affected the bond strength. The greatest degree of conversion was for G2 with Single Bond Universal, significantly higher than Scotchbond and RelyX U200. G4 provided highest value for the cement layer, and statistically higher than G1. G1 showed the highest strength than G3 associated to resin cement or self-adhesive resin cement. Different fracture patterns were observed.**

Conclusions: **Aging methods reduced the bond strength. Degree of conversion was highest in the adhesive layer photo activated and in the cement layer with dual activation. Different fracture patterns were observed.**

Clinical Relevance: **Resin cements degree of conversion and fracture pattern of the hybrid layer are relevant factors in the different bond strength values obtained in adhesive protocols.**

Keywords: Composite resins, dentin bonding agent, indirect restorations, resin luting cement, thermocycling.

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I. Introduction

Fluid infiltration between teeth and indirect restorations has been defined as a clinically undetectable dynamic phenomenon that introduces bacteria, molecules, and ions into the gap between the dental wall and the restorative material [1]. This infiltration can cause tooth hypersensitivity, development of secondary caries and pulp problems [2], [3]; therefore, the maintenance of marginal sealing is considered an important factor for determining the longevity of indirect restorations [4].

In vitro studies show that levels of microleakage differ significantly according to the adhesive cementation technique used [1]. Among the types of cement available, resin cements present the best results; however, all these resin materials allow some level of microleakage [1], [4], [5]. Conventional resin cementation can associate different adhesive systems, enabling the clinical use of diverse combinations between adhesives and resin cements. As such, it is possible to use conventional adhesives for which the cementation technique requires prior etching-and-rinsing of the region to be restored [6] or self-etching methods that require selective enamel etching [7].

Adhesive systems can vary in the number of steps and bottles used [8]; conventional cementation techniques are more complex and are sensitive and susceptible to the dentist's ability as failures may occur during cement application and significantly compromise the bond strength [9]. In an attempt to overcome the sensitivity of the cementation technique due to the number of steps, self-adhesive cements have been developed that do not require any association either with adhesive systems or with any previous treatment of tooth structures [10]. Simplified adhesive protocols provide reduced technical sensitivity; however, they result in greater water permeability and a consequent reduction in bond strength after storage time [11],

[12]. Unlike the adhesive systems that employ 2 or 3 steps [13], these simplified adhesives show less water permeability due to the application of the hydrophobic monomer layer [14], [15].

Another factor related to adhesive layer permeability is the lower degree of conversion of the adhesive monomers, due to incomplete polymerization, which may also contribute to the microleakage of the adhesive systems [16], [17]. Incomplete polymerization of adhesives results in the formation of a porous layer with reduced sealing capacity [18]-[20], shorter restoration durability [21], [22], and impaired material properties [23]. Therefore, higher degrees of conversion are associated with improvements in the mechanical properties of resinous materials [23]-[25], as well as better biocompatibility [26], [27].

The maintenance of cement adhesion to the tooth structure is susceptible to critical situations in the oral environment. Adhesive interface degradation and reductions in resin cement bond strengths are usually related to thermal changes (that can be simulated by the thermal cycling) caused by the different coefficients of linear thermal expansion between composites and tooth structure [28]. In addition, the adhesive interface can also degrade in response to diverse chemical alterations caused by acidic foods and beverages [29]. Thus, evaluating the combination of techniques that result in lower adhesive interface permeability is important for understanding the mechanical behavior of dental materials and predicting the clinical survival of cemented restorations and the maintenance of marginal sealing *in vivo* [30].

A number of adhesive techniques with different levels of application complexity are available, but there is no consensus as to which indirect cementation technique best withstands the challenges present in the oral cavity. The aim of this study was to evaluate the bond strength of cements applied with different adhesive techniques to dentin and characterize the fracture patterns that occur and the degree of conversion of the hybrid layer after aging processes.

II. Materials and Methods

A. *Preparation of Dentine Blocks*

Ninety bovine incisor teeth were used after scraping with a periodontal curette to remove organic residues and polishing with a brush (Microdont, Sao Paulo, SP, Brazil) and pumice paste (SS White, Rio de Janeiro, RJ, Brazil). The teeth were sectioned with a metallographic cutter (Isomet 1000, Buehler, LakeBluff, Il, USA) using a diamond disc in order to obtain dentine blocks with dimensions of 5 x 5 x 2 mm. The dentine block dimensions were standardized with a metallographic polisher (APL-4, Arotec, Cotia, SP, Brazil) using granulation #600 silicon carbide sandpaper (Waterproof sandpaper, Carborundum Abrasives, Sao Paulo, SP, Brazil), and then water cooled, and cleaned with ultrasound (Unique Ultrasonic Cleaner, Indaiatuba, SP, Brazil) for 10 min.

The dentine blocks were stored in distilled water in an incubator (Electrolab Laboratory Equipments; Sao Paulo, SP, Brazil) at 37.5 °C until the moment of use for a maximum period of six months. After storage, blocks were embedded in rigid PVC cylinders with self-curing acrylic resin, and divided into four groups according to the cementation technique used: 1) Adper Scotchbond Multi-purpose Plus conventional three-step adhesive (3M ESPE, St Paul, MN, USA) together with RelyX Ultimate dual-curing resin cement (3M ESPE); 2) Single Bond Universal self-etching adhesive from a single bottle (3M ESPE) and RelyX Ultimate dual-curing resin cement (3M ESPE); 3) RelyX U200 self-adhesive resin cement (3M ESPE), and 4) Multilink A and B two-bottle self-etching primer (Ivoclar Vivadent; Schaan, Liechtenstein) and Multilink Automix dual-curing resin cement (Ivoclar Vivadent).

B. *Cementation Technique*

Silicone matrices (Express XT, 3M ESPE) containing a hole with dimensions of 0.8 mm in diameter and 1.0 mm in depth were fixed with wax (Lysanda, Sao Paulo, SP, Brazil) and used to place the cement on the dentine surface. The cement photo activation was performed using a Radii Cal (SDI, Sao Paulo, SP, Brazil) with a wavelength of 440 - 480 nm and power of 1200 mW/cm^2. A 2.0-mm thick composite resin disk was placed between the silicone matrix and the photo activator tip to simulate the light scattering that occurs during the photo activation of the indirect restoration. After cement polymerization, the matrix was carefully removed with an explorer probe.

The following adhesive cementation techniques were used:

1) The dentine block was etched-and-rinsed with 37% phosphoric acid (Condac; Dentscare, Joinville, SC, Brazil) for 15 s, washed with water for 15 s and the excess water was removed with absorbent paper. The adhesive protocol was as indicated by the manufacturer; a drop of activator was mixed with a drop of primer and applied to the dentine block with a disposable applicator (Microbrush, KG Sorensen, Cotia, SP, Brazil), followed by a light jet of air for 5 s and catalyst application. The dual-curing resin cement (RelyX Ultimate, 3M ESPE) was manipulated, applied to dentine surface and photo activated for 20 s.
2) The self-etching adhesive was actively applied to the dentine surface for 20 s and the excess removed with a disposable applicator, followed by a light jet of air for 5 s. The dual-curing resin cement was manipulated, applied to the dentine surface, and photo activated for 20 s.
3) The moisture excess on the dentine was removed with absorbent paper. The self-adhesive resin cement was then manipulated and applied to the dentine surface. After 3 min, the resin cement was photo activated for 20 s.
4) One drop of Primer A and one drop of Primer B were mixed, and applied on the dentine surface. After 30 s, a light jet of air was administered. The dual-curing resin cement was manipulated according to manufacturer's instructions, applied to dentine surface, and photo activated for 20 s. All samples were stored in distilled water in an incubator (Electrolab) at 37.5 °C for 7 days.

C. *Treatment of the Specimens*

1) *Control group (CG)*

The specimens were analyzed after distilled water storage at 37.5 °C for 7 days.

2) Thermal cycling (TC)

The specimens were subjected to 5,000 thermocycles in distilled water baths at 5 ± 2°C and 55 ± 2°C for 30 s each, with transfer intervals of 30 s using a thermocycle simulator (OMC 300 PLUS; Odeme, Luzerna, SC, Brazil), according to a previous study [31].

3) Chemical aging (CA)

The specimens were stored in 10% sodium hypochlorite solution at room temperature for 1 h, according to dental literature [29].

D. Microshear Test

The specimens were submitted to the micro shear test using a universal testing machine (EZ-test; Shimadzu; Kyoto, Japan) at a cross speed of 0.5 mm/min until failure. The adhesive interface area was measured with a digital caliper (Digimatic; Mitutoyo, Illinois, USA) to calculate the µSBS. The fracture pattern was assessed with a stereoscopic magnifying glass (VMM-100-BT - Walter UHL, Germany) with a 60x magnification and the failures classified as adhesive (dentin/adhesive or adhesive/cement), cohesive (adhesive or cement) or mixed.

E. Degree of Conversion in the Hybrid Layer

Composite resin blocks (Filtek Z350XT; 3M ESPE) were fixed in the dentin blocks (5x5x2 mm) according to the cementation protocols, as previously mentioned. Subsequently, the blocks were sectioned in sticks with approximately 1x1-mm² cross sections for the analysis of the degree of conversion (DC).

Sticks from each block (n=5) were polished with a felt disc and diamond paste, followed by washing under ultrasound for 10 min. Micro-Raman XploRA spectrophotometry (Horiba; Paris, France) was performed with 3.2 mW for HeNe laser potency. A magnification of 100x was used to obtain a spectral area of 1 µm diameter, which was necessary to measure the DC of the hybrid layer and resin cement layer, according to previous methodology [32].

The vibrational spectrum of the unpolymerized adhesives and cements (average of two unmixed cement pastes) was detected in the range 1580-1660 cm^{-1} with peak heights detected at 1610 cm^{-1} (double bond C=C aromatic) and 1640 cm^{-1} (double bond C=C aliphatic methacrylate). The same peaks were detected in the sticks, and the relationship between them was measured to calculate the DC.

The fracture pattern was assessed with a stereoscopic magnifying glass (VMM-100-BT - Walter UHL, Germany) with a 60x magnification and the failures classified as adhesive (dentin/adhesive or adhesive/cement), cohesive (adhesive or cement) or mixed.

F. Statistical Analysis

Data were analyzed by the Kolmogorov-Smirnov statistical test to confirm normal distributions. Bartlett's test was applied to confirm the normality hypothesis of the data. Two-way ANOVA was used to assess differences in bond strength results, and one-way ANOVA for DC data. Tukey's test was applied for multiple comparisons ($\alpha=0.05$). Pearson's test ($\alpha=0.05$) assessed the correlation between DC and µSBS.

III. Results

A. Bond Strength

Fig. 1 shows the mean and standard deviation for the µSBS of the specimens. Statistical analysis showed that the cementation techniques ($p<0.001$) and aging methods influenced the µSBS ($p<0.001$) and that there was significant interaction between both factors ($p<0.001$). Technique 1 demonstrated the highest µSBS values for both the control group and after applying aging methods ($p<0.001$). Technique 2 presented a higher bond strength value in relation to the techniques 3 and 4 when associated with all factors, whereas the techniques 3 and 4 presented statistically similar results. For techniques 2, 3 and 4, the TC aging significantly reduced bond strength when compared to the control group.

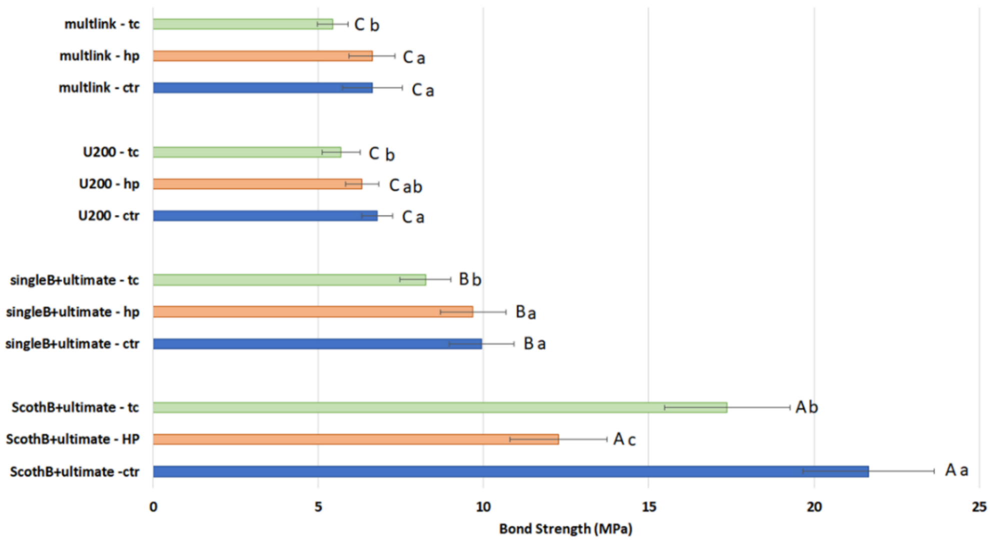

Fig. 1. Bond strength for each group. Capital letters show significance between cementation types, and lowercase letters show significance between aging types for each cementation method.

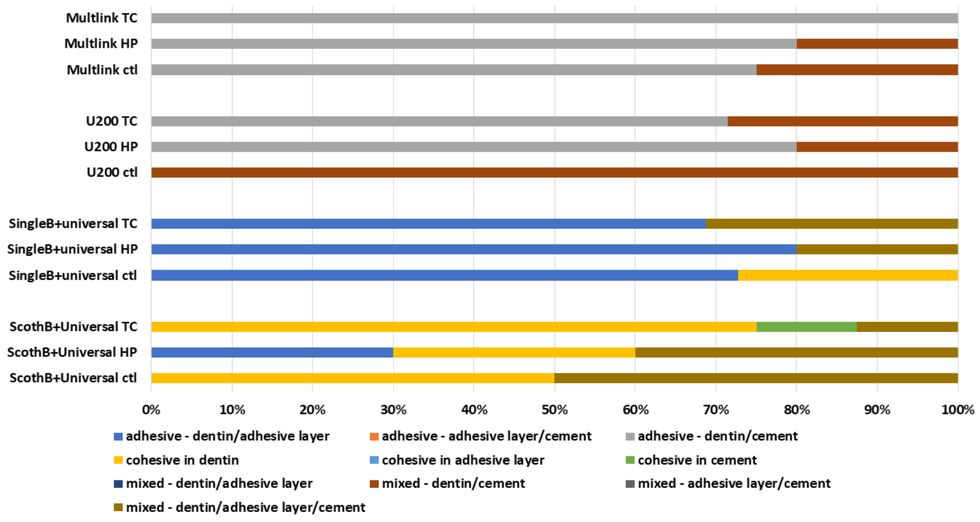

Fig. 2. Fracture pattern for each group after micro shear test.

Fig. 2 shows the fracture patterns for each group. For technique 1, there was a predominance of cohesive fractures in the dentin and mixed fractures. For technique 2, the fracture pattern was predominantly adhesive at the dentin-adhesive interface. For techniques 3 and 4, there was a predominance of adhesive fractures at the dentin-cement interface, with the exception of the control group for the technique 3 which demonstrated a predominance of mixed fractures in the dentin and cement layers.

B. Degree of Conversion

Table I shows the degree of conversion (%) for the adhesive and cement layers. Single Bond Universal (G2) presented the highest degree of conversion in the adhesive layer, while the lowest DC was observed for Adper Scotchbond Multi-purpose Plus Scothbond (G1). Multilink Primer A and B (G4) presented an intermediate value.

In the cement layer, the use of RelyX U200 achieved the highest DC, while the lowest value was observed for RelyX Ultimate together with Adper Scotchbond Multi-purpose Plus. RelyX Ultimate with Single Bond and the Multilink Automix presented intermediate values.

TABLE I: DEGREE OF CONVERSION (%) FOR THE ADHESIVE AND CEMENT LAYERS

Materials	Adhesive Layer
Adper Scotchbond Multi-purpose Plus	46.2(10.7) B
Single Bond Universal	76.3(6.2) A
Multilink Primer A e B	61.6(2.9) AB
	Cement layer
RelyX Ultimate with Adper Scotchbond Multi-purpose Plus	46.3(8.4) B
RelyX Ultimate with Single Bond	52.1(9.4) AB
Multilink Automix	51.1(4.1) AB
U200	65.7(3.8) A

IV. DISCUSSION

Adhesion to tooth dentine is a one of the biggest challenges in adhesive restorations. Several systems are available for adhesive cementation and permit different application protocols. Thus, understanding and characterizing the bonding interface are fundamental factors for achieving success and longevity of the tooth restoration in operative dentistry practice. In this study, the adhesive cementation techniques positively influenced the values of bond strength at the adhesive interface. The adhesive cementation protocol with etching-and-rinsing provided greater bond strength, when compared to simplified cementation techniques that did not demineralize the dentin with phosphoric acid.

The association of the conventional 3-step adhesive (Adper Scotchbond Multi-purpose Plus) with the conventional dual-curing resin cement (RelyX Ultimate) showed the highest μSBS values. Adhesive cementation involving 3-step adhesive systems is considered a gold standard for bonding agents (to enamel and dentin), showing greater bonding effectiveness when compared to self-adhesive, self-etching and conventional two-step systems [6], [33]. Conventional 3-step adhesives, which maintain the etch, primer and adhesive stages in separated steps, permit different therapeutic objectives and better technical control in each step [6]. Self-etching adhesives are available in a single bottle with a mixture of water, solvents and monomers, showing relatively less bond strength and reduced mechanical properties [11], [12], [23].

In contrast, 3-step adhesive systems require more clinical time and show greater technical sensitivity, which can compromise the cementation process and the restoration longevity [34], [35]. Self-etching adhesives promote a lighter demineralization, without removing the smear layer and no tooth sensitivity is expected [36], [37].

One of the clinical advantages of the Scotchbond Multi-purpose Plus Adper (Activator, primer and catalyst) system for indirect cementation, when compared to the photopolymerizable three-step adhesive system, is that the adhesive is not applied to the tooth substrate and does not form an adhesive layer that can interfere in the adaptation of the indirect restoration. When techniques 1 and 2 were compared (same cement), the 3-step adhesive system showed better bond effectiveness, in agreement with the findings of the literature [38]. In contrast, other studies with self-adhesive cements applied to dentin have shown satisfactory adhesion levels that are comparable to multi-bottle adhesives [37].

Technique 3 (self-adhesive resin cement) presented a lower µSBS when compared to the association of the adhesive with cement, as used in techniques 1 and 2. This result was probably due to the fact that all the cements have higher viscosity and greater water permeability, consequently demonstrating reductions in bond strength after water storage [11], [12]. The same does not occur with the adhesive systems with 2 or 3 steps [13], which present lower water permeability due to the application of the hydrophobic monomer layer [14], [15].

The All-in-One cements promote tooth demineralization simultaneously with the infiltration of the smear layer and dentine substrate. Demineralization occurs through the action of functional monomers with light demineralization characteristics, resulting in micromechanical locking, and the formation of a sub-micrometric hybrid layer [39]. Technique 4 showed similar values to those of technique 3. The cementation process with Multilink Primer A and B involves the association of the mixture of the acidic primer and a specific cement for the system. We suggest that the milder demineralization that occurs in techniques 3 and 4 may be responsible for the lower bond strength values due to the limited acidity of the primers. As an analogy, a previous study [40] showed that the removal stress for lithium disilicate crowns after thermocycling did not differ for the associations of Multilink Automix with Multilink Primer and RelyX Ultimate with Scotchbond Universal; however, the removal stress for both cements differed significantly from that for the association of NX3 Nexus with OptiBond XTR 41.

Aging techniques have been shown to degrade the adhesive interface of indirect bonding systems. The thermal cycles promote a reduction in the bond strength of all the systems. This result can be explained by the fact that the thermal cycle accelerates the hydrolysis of non-infiltrated collagen fibrils (unprotected) and the leaching of unreacted monomers [39], [41].

Sodium hypochlorite decreased the bond strength in technique 1 due to the greater amount of collagen fibrils exposed during acid conditioning, leaving the material more vulnerable to chemical degradation and interfering negatively in the adhesive bonding values [42]. Simultaneous dentine infiltration and demineralization occurs in the self-etching technique, leaving a smaller amount of exposed collagen fibrils. This degradation is not enough to impact negatively on the bond strength [29], [42].

Technique 1 showed a predominance of cohesive and mixed fractures in dentin. Previous studies report that these fracture types are due to effective infiltration of the resin monomer to the dentine tissue and formation of a well-defined hybrid layer [43], [44]. Adhesive fractures were predominant at the dentine-adhesive interface for technique 2. Similarly to technique 1, a well-defined hybrid layer and strong chemical bonds were formed between the functional monomer and dental tissue [43], [45]. Conversely, in techniques 3 and 4 there was a greater predominance of adhesive fracture at the dentin-cement interface, except for the control group for technique 3, where dentin-cement mixed fractures predominated, probably due to the lower dentine infiltration caused by these monomeric systems [43], [44].

The analysis of the degree of conversion demonstrated the highest values for the Single Bond Universal adhesive, followed by the Multilink Primer A and B. In the cement layer, U200 showed the highest value, followed by the RelyX Ultimate when associated with the Single Bond Universal, and by Multilink Automix. Similar degrees of conversion were obtained in a previous study for different resin cements [46]. U200 self-adhesive resin cement is formulated with polymerization accelerators, such as p-toluenesulfinic acid, which acts when the pH of the cement is elevated by the neutralization that occurs when it is placed in contact with the tooth substrate to form the inter diffusion layer. This accelerator increases the degree of cement conversion; however, it does not participate in the adhesion process [47]. Dual-curing resin cements showed higher degrees of conversion, when compared to light-cured cements [48].

Another important factor in the cement bonding is the layer thickness of the dental composite. In the current study, a 2 mm-thick composite layer was used; however, it should be taken into consideration that changes in the tooth bond level may occur in thicker restorations due to the lower light passage during the photo activation of the resin materials.

When the values of the degree of conversion were compared with the bond strength values, no correlation was found between these factors. Although there was no direct correlation between the effectiveness of adhesive bonding materials with the degree of conversion, it is still important to remember that the characteristics and properties of resin cements and adhesive systems are dependent on the appropriate level of the degree of conversion [49], as shown in this current study.

V. Conclusion

According to this study, the following conclusions can be drawn:

1) Cementation techniques influenced the µSBS and fracture pattern, and the three-step adhesive when used in association with resin cement showed better bonding effectiveness to dentin, when compared with the self-etching adhesive associated with the resin cement or self-adhesive resin cements or self-etching adhesives.
2) The aging processes reduced the µSBS for all techniques, especially thermal cycling.
3) Hypochlorite accelerated collagen fiber degradation and was more harmful to the etch-and-rinse system.
4) The degree of conversion did not influence the µSBS values of the tested systems.

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Conflict of Interest

Authors declare that they do not have any conflict of interest.

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