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Original Article

Shear Bond Strength and Adhesive Remnant of Three Protocols used for Bonding Orthodontic Brackets

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Abstract

Objective: To test the shear bond strength and the amount of adhesive remnant on the enamel after debonding of a conventional orthodontic composite system, a flowable composite resin, and a self-adhesive resin cement. **Material and Methods:** Thirty extracted bovine incisors were allocated in three groups, according to the type of adhesive: Group XT (Transbond XT), Group FL (Flow Z350), and Group RX (RelyX U100). All groups had etching with phosphoric acid. Groups XT and FL received primer before adhesive. Stainless steel metal brackets were bonded using the respective adhesive. Teeth were submitted to shear bond strength (SBS) test, followed by measurement of adhesive remnant. Intergroup comparison of SBS values were performed by one-way Anova and Tukey post-hoc test. Kruskal-Wallis test and the Mann-Whitney U-test were used to compare adhesive remnant. **Results:** Transbond XT presented higher bond strength than RelyX U100 and Flow Z350; all adhesives exhibited bond failure within the adhesive. All groups showed bond failure occurring within the adhesive. Transbond XT and RelyX U100 left significantly more adhesive remnant on the tooth surface than Flow Z350. **Conclusion:** All three adhesive systems had bond strength above the minimum for clinical routine use. As regards to bond strength, Transbond XT performed better than the resin cement and the flowable resin. Bond failure occurred within the adhesive in all groups.

Keywords: Composite Resins; Dental Bonding; Shear Strength.

Introduction

The development of the acid-etching technique by Buonocore [1] and subsequent modifications [2,3] to improve bonding technique for orthodontic purposes made bond strength high enough to replace banding of orthodontic attachments. Acid-etching of enamel increases bond strength by producing a micromechanical union between the adhesive and the enamel. Currently, two main strategies are used to create long-lasting enamel bonding: (1) the 3-step technique – acid etching followed by a resin thin layer which penetrates the etched enamel before the bracket coated by a composite is bonded, which is more widely used [4], and (2) self-etching systems or acidic primers combining the etching and priming steps, which minimize the working time during bonding and eliminates possible damage to the gingival tissue [5].

Orthodontic conventional composites, flowable composites, glass ionomer cements, and, more recently, resin cements have been used for bonding orthodontic attachments [6]. Flowable composite resins present some advantages over conventional composites because of their clinical handling characteristics, such as no stickiness and fluid injectability [7]. Additionally, it leaves thinner adhesive excess around brackets, called flashes, than conventional composite resins. Thick flashes need to be removed prior to adhesive cure in order to prevent the adhesive from causing mechanical irritation to the gingiva [8] and to decrease the incidence of plaque accumulation and subsequent enamel demineralization [9]. Flowable resins showed bond strength to metallic brackets similar to a conventional orthodontic composite resin when using etching and primer before bonding [10].

The capacity of resin cements of forming chemical union with enamel, dentin, ceramic, metals, and composite materials [11] could present better performance of bonding than composites, which has bond strength to the bracket and to the enamel produced by mechanical unions. Moreover, some resin cements called self-adhesive require no etching, priming or bonding agents to bond to the tooth surface. Therefore, they are more likely to reduce time required for bonding. It was previously shown that Rely X Unicem, a self-adhesive resin cement, when applied without previous etching, showed bond strength level considered clinically acceptable, despite its shear resistance was lower than conventional orthodontic composites [6]. It is suggested that pre-etching of enamel with phosphoric acid before bonding could increase bond strength of this cement because the higher content of minerals in enamel compared to dentin requires a stronger acid [12]. Even requiring acid-etching of enamel, the self-adhesive resin cements would still reduce the step of priming, compared to conventional composites. However, no studies are available on the bond strength of resin cements on previously etched enamel surfaces.

During debonding, failure can occur at the adhesive-bracket interface, at the adhesive-enamel interface, and/or within the adhesive (cohesive failure). If a strong bond to the enamel has been achieved, it is preferable that bond failure occurs at the adhesive-bracket interface in order to prevent tearing the enamel [13], despite more adhesive remnant on the tooth surface might increase time required for adhesive cleanup after debonding [14].

Bond strength and failure mode upon debonding are relevant factors considered by a practitioner when choosing an adhesive for bracket bonding. Accordingly, the aims of this study were to test the shear bond strength and the amount of adhesive remnant on the enamel after debonding of a conventional orthodontic bonding system, a flowable composite resin, and a self-adhesive resin cement on previously etched enamel.

Material and Methods

Experimental Design

This *in vitro* study evaluated (1) *shear bond strength* of orthodontic adhesives and (2) the amount of *adhesive remnant* of each system after debonding, at three levels – a light-cured orthodontic conventional composite resin Transbond XT (3M Unitek, St. Paul, USA), a light-cured nanofilled flowable resin Flow Filtek Z350 XT (3M Unitek, St. Paul, USA), and a dual-cured self-adhesive resin cement Rely-X U100 (3M Unitek, St. Paul, USA). Table 1 shows further details of each group and adhesives used.

Table 1. Materials used in this study.

Classification	Trade name (Manufacturer)	Composition (% by weight)*	Application mode
Acid-etching agent	Alpha Acid (DFL, Rio de Janeiro, RJ, Brazil)	Phosphoric Acid (37%), Colloidal Silicon Dioxide (Aerosil 200), Methylene Blue CI 52015 and Deionized Water.	
Group XT Conventional orthodontic adhesive	Primer and adhesive: Transbond XT (3M Unitek, St. Paul, MN, USA)	Primer: Bis-GMA (45–55%), TEGDMA(45–55%), 4-(dimethylamino)-benzeneethanol (<.5%), DL-camphorquinone (<.3%), hydroquinone (<.03%) Adhesive paste: Bis-GMA (10–20%), Bis-EMA (5–10%), silane treated quartz (70–80%), silane treated sílica (<2%), diphenyliodonium hexafluorophosphate (<.2%)	1. Enamel etching 2. Primer 3. composite+Bracket Photoactivation
Group FL Nanofilled flowable composite resin	Primer: Single bond (3M ESPE, St Paul, MN, USA) Adhesive: Filtek Z350 (3M ESPE, St Paul, MN, USA)	Primer: Ethyl alcohol (25–30%), silane treated silica (nanofiller) (10–20%), Bis-GMA (10–20%), HEMA (5–10%), glycerol 1,3-dimethacrylate (5–10%), copolymer of acrylic and itaconic acids (5–10%), water (<5%), UDMA (1–5%) Adhesive: Bis-GMA (10–15%), TEGDMA (10–15%), UDMA (1–5%), silane-treated ceramic (52–60%), silane-treated silica (3–11%), silane-treated zirconium oxide (3–11%), functionalized dimethacrylate polymer (1–5%)	1. Enamel etching 2. Primer 3. Flowable resin +Bracket Photoactivation
Group RX Self-adhesive resin cement	RelyX U100 (3M Unitek, St. Paul, USA)	Base: glass poder (55-65%), methacrylate phosphoric acid esters (15-25%), dimethacrylates (10-20%), silane treated silica (1-5%), sodium persulfate (1-5%), Catalyst: glass powder (55-65%), dimethacrylates (20-30%), silane treated silica (1-5%), p-toluene sodium sulfate, calcium hydroxide.	1. Enamel etching 2. Resin cement +Bracket Photoactivation

*Composition of materials according to information obtained from the manufacturers. Bis-GMA: Bisphenol A-Glycidyl Methacrylate; TEGDMA: Triethylene Glycol Dimethacrilate; Bis-EMA: Bisphenol A-Bis(2-Hydroxyethyl Ether) Dimethacrylate; UDMA: Urethane Dimethacrylate.

Sample Preparation

Thirty bovine incisors were collected from local authorized slaughterhouses, washed in running water, and stored in a 0.5% thimol solution at room temperature. The solution was renewed weekly. The inclusion criteria included newly extracted teeth, good preservation status, and no damage from the extraction process.

All teeth were freed from remnants of periodontal ligament, and had their roots cut at 4 mm apically to the buccal cementoenamel junction by a flexible diamond disc (KG Sorensen, Cotia, Brazil) attached to a low-speed handpiece. After receiving grooves on the roots using carborundum disc in order to create mechanical retentions, the incisors were inserted in polyvinyl chloride rings (20 x 20mm) fabricated and filled up with liquid polystyrene resin. The roots were completely embedded into the resin in the center of each ring, leaving only the crowns exposed. With the aim of standardizing the tooth inclination, a geometry acrylic triangle was used to keep their buccal crown surfaces perpendicular to the resin base. The rings were removed after polystyrene cure and the specimens stored in distilled water.

Adhesive Procedures

The buccal crown surface of each tooth was rinsed for 30 seconds and dried after a 10-second polish with fluoride-free pumice slurry and rubber cups. Subsequently, the area receiving the brackets was acid-etched for 15 seconds with 37% phosphoric acid gel Alpha Acid (DFL Ind. Com. Ltda, Rio de Janeiro, RJ, Brazil) applied with a syringe. The teeth were rinsed with water for 30 seconds and dried for 20 seconds until observing a chalky appearance. Teeth were allocated in three groups, as described in Table 1.

Group XT (Transbond XT) received Transbond XT primer whilst group FL (Flow Z350) received Single Bond primer before bracket bonding with the main adhesive. Primer was spread on the acid-etched enamel surface using a brush-like applicator, air dried for 10 s (20 cm), and then photoactivated for 10 s with a light emitting diode (LED) Radii plus (SDI Limited, Victoria, Australia). Group RX (RelyX U100) did not receive primer.

Stainless steel metal brackets for lower incisors Roth prescription (Eurodonto, Curitiba, PR, Brazil) were directly bonded to the prepared enamel. The adhesives were placed in the center of each bracket base and homogeneously distributed with a composite spatula. The brackets were pressed firmly against the tooth surface in order to assure a thin layer of adhesive and to standardize the procedure. After removing the flash around the base, the adhesives were light-cured for 20 s (10 s on each side) using a LED device, according to the manufacturer's instructions.

After bonding, the specimens were stored in distilled water at 37°C for 24 hours, before shear bond strength analysis.

Shear Bond Strength Analysis (SBS)

A universal testing machine Autograph AG-IS (Shimadzu, Tokyo, Japan,) with a one-KN load cell (Shimadzu) was used to perform the SBS test. A crosshead speed of 0.5mm/min was used.

This machine was calibrated every five tests. Samples were adapted into a metal clamp in a position favoring the application of a shearing movement parallel to the buccal surface of the crowns. The apex of a triangle made of stainless steel rectangular wire (0.019 x 0.025 inch) was attached to the machine while its base was encased under the lower wings of the brackets to deliver the shearing stress. Shear strength values were obtained in Newtons (N) and then converted into mega Pascals (MPa).

Measurement of Adhesive Remnant

After debonding, each specimen was positioned beside an endodontic ruler (SS White, Lakewood, USA) under an optical microscope (16x magnification) and photographed by a digital camera D70S (Nikon, Melville, USA) with macro lens 105mm (Sigma, Rokonkoma, USA) in ImageJ software (NIH, Bethesda, USA) the scale of each image was set using the distance shown at the ruler as reference, followed by measurement of the area of the residual composite remaining on the teeth surfaces, as described in Figure 1. The percentage of the remnant adhesive was calculated by the area of the remnant adhesive (mm^2) divided by the area of the bracket base ($8.8mm^2$) multiplied by 100. Each tooth was scored with the adhesive remnant index (ARI), according to the amount of material remaining on the enamel surface as follows: 0 = no adhesive remaining, 1 = less than 50% of the adhesive remaining, 2 = more than 50% of the adhesive remaining, and 3 = all adhesive remaining with a distinct impression of the bracket base.

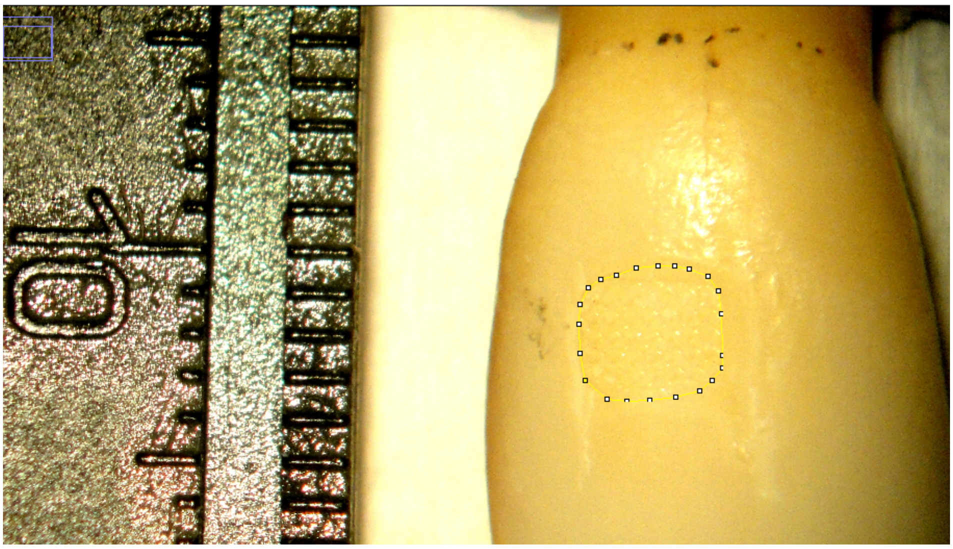

Figure 1. Measurement of the area of remnant adhesive on the tooth surface using ImageJ (NIH, Bethesda, USA).

Statistical Analyses

The Kolmogorov-Smirnov test was applied to data of the SBS and the area of adhesive remaining on tooth. SBS showed normal distribution in each group and one-way analysis of variance (ANOVA) followed by the Tukey test were used for intergroup comparison of the SBS values. As the data for adhesive remnant area did not show normal distribution in the groups, they were compared using the Kruskal-Wallis test and the Mann-Whitney U-test. Significance for all statistical tests was set at P<.05. All data were entered and analyzed by Statistica 7.0 (Statsoft, Tulsa, OK, USA).

Results

Descriptive analysis of SBS, including mean and standard deviation, and intergroup comparison of SBS values are described in Table 2. Transbond XT and RelyX showed clinically acceptable SBS. The one-way ANOVA (P= 0.00) followed by Tukey tests showed that Transbond XT presented statistically significant higher SBS than Rely X (P= 0.022) and Flow Z350 (P= 0.00).

Table 2. Comparison of shear bond strength values (MPa) obtained for tested groups, analyzed by ANOVA followed by Tukey tests.

Groups	Mean (SD)
Transbond XT	10.3 (1.5) [a]
Flow Z350	4.1 (1.5) [b]
Rely-X U100	6.5 (1.6) [b]

Groups marked by different superscripted letters showed significant differences with one another at p<0.05.

Values for ARI scores and percentage of area of adhesive remaining on tooth surface are shown in Table 3. The Kruskal-Wallis test and the Mann Whitney *U*-test indicated that the area of adhesive remnant presented by Flow Z350 was significantly smaller (P < 0.05) than the values shown by Transbond XT and RelyX. These two groups exhibited similar areas of adhesive remnant (*P*= 0.65). According to ARI scores, 70% of specimens of group XT and 90% of group RX had more than half area of adhesive remaining on the tooth surface after bracket removal. Contrarily, only 40% of specimens in group FL left more than half of adhesive area on the enamel.

Table 3. Frequency distribution of the Adhesive Remnant Index (ARI) scores and the mean percentage (SD) of area of adhesive remaining on the tooth surface in each group.

ARI	Transbond XT		Flow Z350		RelyX U100	
	Number	% bracket area (SD)	Number	% bracket area (SD)	Number	% bracket area (SD)
0	0	0.0	0	0.0	1	0.0
1	3	27.7 (9.9)	6	24.6 (16.9)	0	0.0
2	6	79.9 (14.2)	4	61.2 (7.4)	9	75.7 (9.9)
3	1	100	0	0.0	0	0.0
Total[1]	10	64.23 (28.13)[a]	10	39.2 (23.1)[b]	10	68.1 (25.7)[a]

[1]Kruskal-Wallis test and Mann-Whitney *U*-test compared the area of adhesive remaining on the tooth surface among groups. Groups marked by different superscripted letters showed significant differences with one another at P<0.05.

Discussion

The results of our research showed that the self-adhesive resin cement (RelyX) and the nanofilled flowable composite (Flow Z350) presented statistically lower SBS than the conventional orthodontic composite (Transbond XT). The higher filler content of Transbond XT might have contributed to this result. A few studies showed that Transbond XT had better performance on higher flexural modulus and SBS values [15,16], and lower contraction stress than less filled adhesives [15]. Very few data of resin cements regarding bracket bonding is available. One study compared the bond strength of RelyX Unicem, without previous etching of the tooth enamel, to conventional orthodontic composites and found SBS about 4MPa lower than Transbond XT [6]. In our study, RelyX U100 also showed lower SBS than Transbond XT, even applied on an acid-etched enamel. This means that enamel conditioning did not increase bond strength of RelyX. Another study observed that bond strength values obtained with Panavia resin cement were lower than orthodontic adhesive system [17], while a different research found no significant difference between Panavia and conventional orthodontic adhesive [18].

Despite SBS values for RelyX were lower than that for Transbond XT, they were greater than the 5.9 to 7.9 MPa considered suitable for routine clinical use [19]. Moreover, SBS values recorded in vivo flowing an orthodontic treatment are significantly lower than those recorded in vitro. The shorter time required for bonding brackets promoted by RelyX could make this adhesive a better option than the conventional 3-step orthodontic composite. However, there is no data comparing the time required for bracket bonding with both systems.

According to the optical microscopic observation, 93% of the sample exhibited debonding within the adhesive (ARI= 1-2). Therefore, all adhesives tended to present bond failure patterns favorable to enamel preservation. Previous studies have found similar bond failure patterns for all three adhesive systems [6,10]. The percentage of tooth surface area occupied by adhesive indicated that Flow Z350 was the system that left less adhesive on the dental structure after debonding, while Transbond XT and RelyX had similar results. Some authors [6] found significant less adhesive remnant for RelyX Unicem than Transbond XT and attributed those results to the lack of acid-etching of the enamel surface. Our results for adhesive remnant of RelyX might have higher due to the enamel acid-etching.

Although the bond strength was not measured under oral conditions, where there are mechanical impact and biochemical changes, in vitro shear debonding forces are considered an acceptable method to evaluate future in vivo comparative conditions [20].

Conclusions

• All three adhesive systems had SBS values above the minimum for clinical routine use, but Transbond XT presented the highest bond strength over RelyX U100 and Flow Z350;

• All adhesives exhibited a cohesive bond failure pattern, which occurs within the adhesive;

• Transbond XT and RelyX U100 left significantly more adhesive remnant on the tooth surface than Flow Z350.

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