

## Evaluation of Bonded Orthodontics Brackets Using Different Adhesive Systems after a Cariogenic Challenge

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### Abstract

**Aim:** The aims of this study were to evaluate the prevention of enamel demineralization and the shear bond strength (SBS) of orthodontic brackets bonded with fluoride and no fluoride conventional and self-etching adhesives and to analyze the characteristics of enamel near the bond area using a polarized light microscope (PLM) following demineralization and remineralization cycling (Des Re).

**Methods and Materials:** Fifty bovine incisors were selected and divided into five groups according to the adhesive system used during the bonding process: G1, Transbond™ XT Adhesive; G2, Single Bond 2 Adhesive; G3, Optibond Solo Plus; G4, Clearfil SE Bond; and G5, Clearfil Protect Bond. Transbond™ XT was used to fix the brackets to the teeth in all groups. After bonding, the groups were separated into cycling and control subgroups. The specimens were submitted to SBS testing and evaluated under a PLM. The results were submitted to ANOVA and Tukey's post hoc tests ( $p < .05$ ).

**Results:** There were no significant differences for SBS after Des-Re cycling. The Clearfil Protect Bond showed the SBS to be statistically lower than the other adhesives used for the control groups. After a cariogenic challenge, the Single Bond adhesive showed an SBS significantly lower than Transbond XT. The Des-Re cycling increased the enamel demineralization induced after the cariogenic challenge.



**Conclusions:** The cariogenic challenge did not reduce the SBS. Optibond Solo Plus and Transbond™ XT adhesives presented the highest SBS while Clearfil Protect Bond had the lowest. The PLM showed that the cariogenic challenge increased the enamel demineralization for all adhesives evaluated, independent of the presence of fluoride.

**Clinical Significance:** An alternative material with the ability to prevent enamel demineralization should be used in orthodontic patients due to the

higher accumulation of plaque around orthodontic brackets.

**Keywords:** Adhesive, orthodontic brackets, shear strength, polarized light microscopy

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## Introduction

Bonding of orthodontic brackets represents one of the greatest advances in orthodontic treatment since it decreases the chair time required for bracket placement. The enamel at the site of bracket placement is acid etched to create a porous surface; then an adhesive resin is applied to create micro-mechanical retention between the resin and porous dental substrate. The acidic monomers of the self-etching adhesives perform the etching and infiltration functions simultaneously to simplify the process.<sup>1</sup> Self-etching primers (SEP) have been used successfully during bonding to reduce technique sensitivity while minimizing the etching of enamel. Although serving the same purpose, SEPs differ in acidity and aggressiveness.<sup>2</sup> However, the bond strength between the self-etching adhesives and enamel is deficient compared to the conventional acid etching technique.<sup>3</sup>

In spite of the progress of *in situ* and *in vivo* studies in cariology, laboratory tests are still



widely used to evaluate dental caries. These are mainly used to determine the effect of fluoride (F) on inhibition of enamel-dentin demineralization and enhancement of remineralization. Among these protocols, there is a consensus that pH-cycling models can be used because they simulate caries development *in vivo*.<sup>4</sup> Nevertheless, before using this method to estimate the anti-caries potential of fluoride products, these models require prior validation in terms of dose-response.<sup>5</sup>

Accumulation of plaque and colonization of important periodontopathic and superinfecting bacteria occur around orthodontic brackets because of the difficulty patients experience in maintaining a favorable level of oral hygiene. Plaque accumulation can result in more inflammation and gingival bleeding as well as dental caries in these areas in patients receiving orthodontic treatment.<sup>6</sup> Caries development occurs due to a lowering of the pH of the biofilm over the dental tissue.<sup>7</sup> Fluoride inhibits the progress of carious lesions by interfering with the demineralization dynamic of lesion formation. As a result, some materials have been developed with fluoride included in their composition, including adhesive systems. Previous studies reported some adhesives may release fluoride ions that are effective in the prevention of cavity wall lesions and the inhibition of secondary caries.<sup>8-10</sup>

The fluoride adhesives play a role in caries inhibition, due to proximity of areas with biofilm accumulation. For caries induction, the dynamic pH cycling through immersion in demineralization and remineralization solutions is the closest *in vitro* method to simulate the development of *in vivo* caries lesions.<sup>4,11</sup> This model simulates the dynamic physical-chemical stages of demineralization and remineralization, with the resulting histological lesions being very similar to the those found *in vivo*. A good method to evaluate the behavior of the enamel after exposure to acid solutions is through the use of a polarized light microscope.<sup>9</sup> Thus, the aims of this study were to evaluate the prevention of enamel demineralization near the bond area using a polarized light microscope (PLM) following demineralization and remineralization cycling (Des Re) and the shear bond strength of orthodontic brackets bonded with fluoride and no fluoride conventional and self-etching adhesives.

## Methods and Materials

### Sample Selection and Preparation

A total of 50 bovine incisors free of caries, cracks, and fractures of the enamel were used. The teeth were washed in water and frozen until the onset of the experiment. The crowns were sectioned and embedded in PVC molds with polystyrene resin prior to bracket bonding. The buccal surfaces were cleaned and polished with a rubber cup and a slurry of pumice and water, followed by rinsing with a water spray and drying with compressed air. The specimens were divided into five groups, according to adhesive systems used for bracket bonding: Transbond™ XT Primer, Single Bond 2, Optibond Solo Plus, Clearfil SE Bond, and Clearfil Protect Bond (Table 1).

### Bracket Bonding

The following procedures were used for bonding the orthodontic brackets to enamel surfaces of the five groups of teeth.

- **Group 1:** acid etching with phosphoric acid 37% for 30 s and application of Transbond XT Primer.
- **Group 2:** acid etching with phosphoric acid 37% for 30 s and application of Single Bond 2.

- **Group 3:** acid etching with phosphoric acid 37% for 30 s and application of Optibond Solo.
- **Group 4:** application of the self-etching primer Clearfil SE Bond for 20 s.
- **Group 5:** application of the self-etching primer Clearfil Protect Bond for 20 s.

After application of the adhesive agent, Transbond XT resin composite was used to bond the brackets to the teeth in all groups by placing the composite on the teeth and placing the brackets (Morelli, Roth, Sorocaba, Sp, Brasil) on the material and pressing them into place to eliminate any excess composite.

Photoactivation was performed for 10 seconds for each side of the brackets with a halogen lamp XL 2500 (3M Dental Products, Monrovia, CA, USA) at a light intensity of 700 mW/cm<sup>2</sup>.

### The pH-Cycling Model

Twenty-five samples were stored in distilled water at 37°C for eight days. The other samples were submitted to a pH-cycling model designed to simulate a cariogenic challenge.<sup>12</sup> Each pH cycling was performed by immersion of the selected samples in a demineralization solution for 7 hours, followed by immersion in a remineralizing solution

**Table 1. Composition of the adhesive systems used in this study.**

Adhesive System	Composition*	Manufacturer	Fluoride
Transbond XT Primer	TEGDMA, BISGMA	3M ESPE	No
Adper Single Bond 2	Bis-GMA, HEMA, GDMA, UDMA, acrylic/itaconic acids copolymers, ethanol, water, 5nm silane treated colloidal silica	3M ESPE	No
Optibond Solo	<i>Adhesive:</i> Bis-GMA, HEMA, GDMA, GPDM, ethanol, CQ, ODMAB, BHT, filler (fumed SiO <sub>2</sub> , barium aluminoborosilicate, (Na <sub>2</sub> SiF <sub>6</sub> ), coupling factor A174 (approximately 15wt% filled)	Kerr	Yes
Clearfil SE Bond	<i>Primer:</i> MDP, HEMA, hydrophilic dimethacrylate, photo-initiator, water <i>Bond:</i> MDP, HEMA, Bis-GMA, hydrophobic Dimethacrylate, photo-initiators, silanated Colloidal silica	Kuraray	No
Clearfil Protect Bond	<i>Primer:</i> MDPB, MDP, HEMA, hydrophilic dimethacrylate, photo-initiator, water <i>Bond:</i> MDP, HEMA, Bis-GMA, hydrophobic dimethacrylate, photo-initiators, silanated colloidal silica, surface-treated NaF	Kuraray	Yes

\*According to the manufacturer.

for 17 hours. The demineralizing solution was composed of acetic acid with 2.2 mM of calcium chloride ( $\text{CaCl}_2$ ); 2.2 mM of sodium phosphate ( $\text{NaH}_2\text{PO}_4$ ); 50 mM of acetate; and 1 ppm of fluoride with a pH of 4.5. The remineralizing solution was composed of 1.5 mM of calcium chloride ( $\text{CaCl}_2$ ); 0.9 mM of sodium phosphate ( $\text{NaH}_2\text{PO}_4$ ); and 0.15 mM of potassium chloride (KCl) with a pH of 7. The remineralizing solution was renewed every day while the demineralizing solution was changed after the fourth day of pH cycling.

### Shear Bond Strength Testing

The shear bond strength (SBS) was performed in a universal testing machine Instron 4411 (Instron Inc., Buckinghamshire, UK), at a crosshead speed of 0.5 mm/min in an incisal-gingival direction, and the maximum stress until failure was recorded. The specimens were mounted in a shear testing apparatus to measure the debonding force in pull mode.

### Polarized Light Microscope

After the SBS evaluation, a qualitative examination using a polarized light microscope (PLM) was performed. Representative samples of each group were cut in sections (slabs) of approximately 400- $\mu\text{m}$  thick (Figure 1) using a diamond saw mounted in an Isomet 1000 (Buehler, Lake Bluff, IL, USA).

The thickness of each section was measured using a digital micrometer with a 1-mm resolution. The PLM evaluation was carried out using a Leica DMLSP polarized light microscope (Leica

Microsystems, Heerbrugg, Switzerland). The images were captured at 20x magnification and transferred to a computer using Image-Pro<sup>®</sup> Plus Version 4.1 for Windows<sup>™</sup> software (Media Cybernetics, Silver Spring, MD, USA).

### Statistical Analysis

The data were submitted to two-way ANOVA and Tukey's post hoc tests. Significance was established at  $p < 0.05$ . After the SBS testing was performed, the qualitative analysis using the PLM was done.

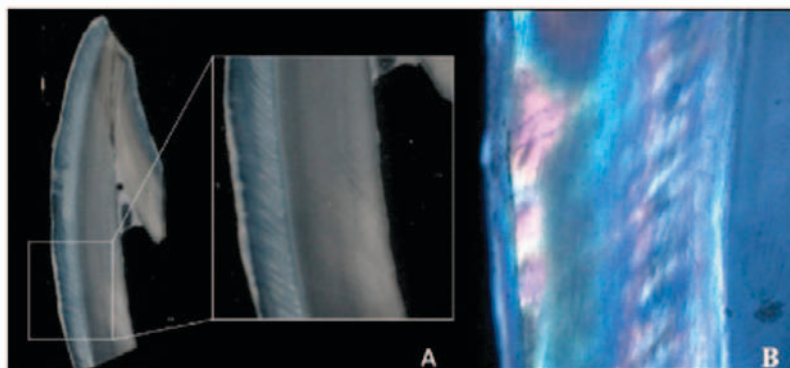
## Results

The SBS data are presented in Table 2.

The results showed that Clearfil Protect Bond presented an SBS significantly lower than other groups ( $p < .05$ ). However, after a cariogenic challenge, Single Bond 2 presented an SBS significantly lower than Transbond XT ( $p < .05$ ), with no significant differences among the other groups.

After a cariogenic challenge, Adper Single Bond 2 presented an SBS significantly lower than the control ( $p < .05$ ), with no significant differences among other adhesives ( $p > .05$ ).

The PLM evaluation of the control group samples (without a cariogenic challenge) showed the integrity of enamel to be unchanged with no evidence of demineralization (Figure 2). However, the samples submitted to cariogenic challenge presented demineralization of



**Figure 1. A.** Image of dental slabs. **B.** Image obtained under a polarized light microscope (20x magnification).

**Table 2. Means (standard deviation) of shear bond strength for different adhesives (MPa).**

Groups	Control		Cariogenic Challenge	
Optibond Solo Plus	26.76 (10.62)	a, A	23.50 (8.54)	ab, A
Transbond XT	25.16 (7.55)	a, A	25.89 (8.58)	a, A
Single Bond	22.00 (6.65)	a, A	12.90 (3.05)	b, B
Clearfil SE Bond	19.68 (7.17)	a, A	15.20 (5.30)	ab, A
Clearfil Protect Bond	14.93 (3.95)	b, A	15.87 (4.88)	ab, A

Means followed by different small letter in the column and capital letter in the row represent statistical differences ( $p < 0.05$ ).

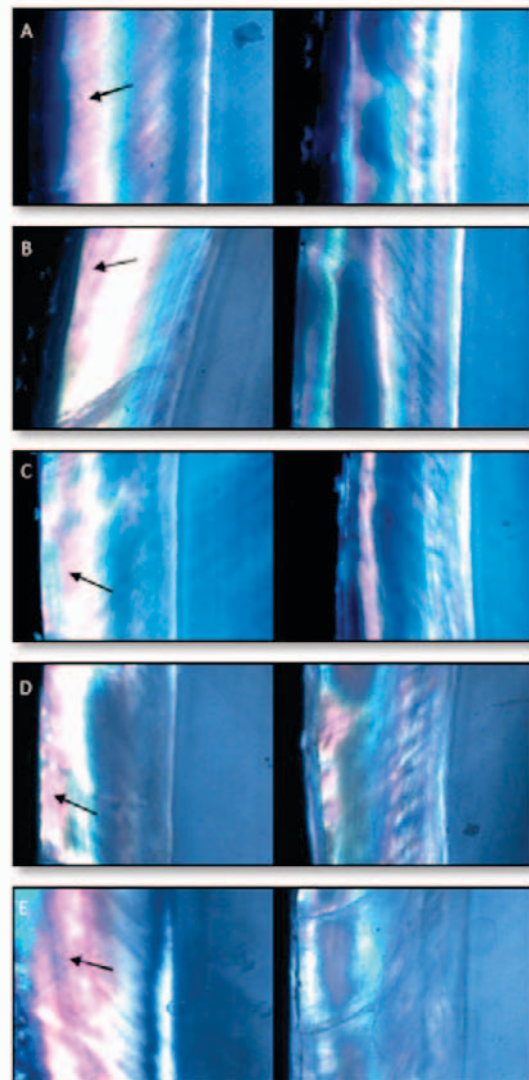
enamel independent of adhesive system. Demineralization zones for all samples submitted to cariogenic challenge are shown in Figure 2 (arrows). Some demineralization zones may be observed in samples of control groups due to acid etching with phosphoric acid. The arrow in Figure 2E identifies a demineralization zone near the adhesive interface.

## Discussion

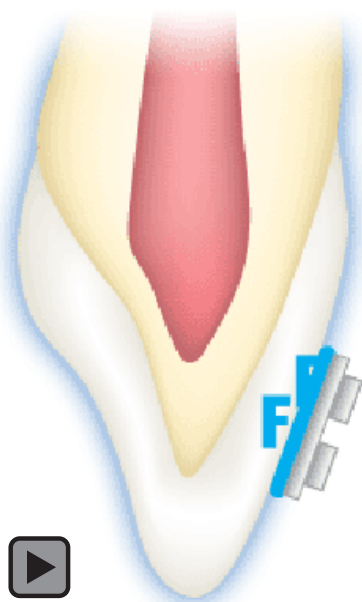
Regular exposure of enamel to various forms of topical fluoride has a greater effect by preventing enamel demineralization rather than the remineralizing of existing lesions because it decreases the solubility of minerals in the enamel crystal lattice in the presence of acid with fluoride ions acting as a catalyst for mineral formation.<sup>13</sup> The use of topical fluoride in its various forms (toothpaste, mouth rinse, gels, varnishes, fluoride-releasing cements) has been the most commonly used caries preventive protocol during orthodontic treatment for at-risk patients, in addition to patient education and regular hygiene visits.<sup>14</sup>

Fluoride-releasing bonding agents were developed to allow the compliance-free, constant exposure to topical fluoride. The use of glass ionomer cement shows a reduction of enamel demineralization when compared with composite resin cements.<sup>15</sup> Although a significant increase in the concentration of fluoride in enamel has been found adjacent to glass ionomer cement, the clinical significance of this increase and the mechanism by which fluoride moves from the glass ionomer cement into the enamel remains unclear.<sup>16</sup>

The images from the PLM showed that restorative



**Figure 2.** Representative images of a control group sample (right) showing lack of caries lesion (demineralization) and images of carious lesion on a sample submitted to pH cycling (left). Lesion aspect on samples bonded with **A.** OptiBond Solo; **B.** Transbond XT Primer; **C.** Adper Single Bond 2; **D.** Clearfil SE Bond; **E.** Clearfil Protect Bond.



systems were unable to prevent the development of demineralized lesions regardless of any fluoride released or any dentin bond adhesive used to fix orthodontic brackets in place. This is in agreement with a previous study<sup>17</sup> that found that fluoride-releasing material reduced demineralization on enamel but was unable to prevent the development of lesions.

This raises the question as to whether the amount of fluoride released by orthodontic adhesives is sufficient to prevent decalcification. One study<sup>18</sup> demonstrated an adhesive that releases fluoride at a level of as little as 0.5 to 1.0 g per square centimeter per day reduced white spot demineralization by 38% over 38 days in rats on a cariogenic diet. The frequency of fluoride application seems to be more important than its concentration. Protection of the enamel can be the result of a slow release of low concentrations of fluoride ions.<sup>19</sup> It is also possible that the protection of the enamel also can be achieved by the initial changes induced by the first release of relatively high concentrations of fluoride ions, or a combination of both processes.<sup>20</sup> There are only a few studies about the fluoride self-etching adhesives used in orthodontics. The literature shows deficient bond strength results of one-bottle adhesives in comparison to two-step adhesives.<sup>21</sup>

The PLM is a useful instrument to verify the effect fluoride has on the demineralization and remineralization processes.<sup>10</sup> In this study,

the PLM was used to observe and verify the qualitative effect of fluoride adhesive after a cariogenic challenge. These observations showed that the fluoride from the adhesives was not able to inhibit the demineralization process. The De/Re solutions show very low concentrations of fluoride ions around the restorations fabricated using fluoride adhesive systems. The low fluoride concentration released from adhesive systems may be justified by the fact that the fluoride ions would probably be confined in the adhesive polymerized resin matrix and/or at the hybrid layer and therefore would not be released to the environment. Since the fluoride in adhesive systems is surrounded by resin matrix, its contact with water would be restricted since its movement may be limited by the matrix itself.<sup>22</sup> However, further investigation is required in order to fully understand the mechanism of fluoride release from adhesive materials.

Only a fluoride-containing adhesive associated with a restorative material also containing fluoride has been found to result in a reduction of artificial secondary caries depth due to the high concentration of fluoride presented in the restorative material.<sup>8</sup> The same study found that the same adhesive system used with a restorative material without fluoride was not effective against caries reduction.

Studies have shown that fluoride-containing restorative materials help prevent secondary caries.<sup>8,23</sup> Nevertheless, the role of fluoride in adhesive systems remains unclear as fluoride-containing adhesive systems had a minimal effect upon the chemically induced secondary caries process and on the bond strength to dentin.

The SBS data are presented on Table 1. The results showed that Clearfil Protect Bond presented a shear bond strength significantly lower than other groups ( $p < .05$ ). However, after a cariogenic challenge, Single Bond 2 presented an SBS significantly lower than Transbond XT ( $p < .05$ ), with no significant differences among the other groups.

Reynolds<sup>24</sup> found that minimum SBS for orthodontic bonding is between 6 and 8 MPa. The lowest found in the present study was 14.93 MPa. The results show that all adhesives tested presented an SBS higher than 14.93 MPa, indicating suitability for clinical use. In addition,

the antibacterial and fluoride-releasing effects of Clearfil Protect Bond would further contribute to the long-term clinical benefits of using this material.<sup>25</sup>

The present study shows that fluoride-containing adhesive systems had a minimal effect upon the chemically induced secondary caries process and on the SBS. Further investigation is required to evaluate the effect of fluoride within the context of a long-term cariogenic challenge as well as *in vivo* or *in situ* studies to evaluate their behavior in intraoral conditions.

## Conclusions

Based on the findings of this study, it can be concluded that:

1. The cariogenic challenge did not reduce the SBS of the adhesives tested. The Optibond Solo Plus and Transbond™ XT adhesives presented the highest, and Clearfil Protect Bond presented the lowest, SBS means.
2. The cariogenic challenge increased the enamel demineralization for all adhesives evaluated, independent of the presence of fluoride.

## Clinical Significance

An alternative material with ability to prevent enamel demineralization should be used for luting orthodontic brackets in orthodontic patients due to the higher accumulation of plaque around orthodontic brackets.

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