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Shear bond strength of an orthodontic self-etching adhesive after intracoronary bleaching

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Structured Abstract

Objectives – To evaluate the effects of intracoronary bleaching on the bond strength of orthodontic brackets using self-etching and total-etch adhesive systems.

Material and Methods – In 60 bovine incisors, a coronal lingual access was made to clean the pulp chamber and standardise the thickness of the dentine. The sample was randomly divided into four groups (each $n = 15$): (CT), control group, without bleaching and bonded with the total-etch system (Transbond™ XT-3M); (CTSE), without bleaching treatment and bonded with the self-etching system (Transbond™ Plus Self-Etching Primer-3M); (BT), treated with 35% hydrogen peroxide for internal bleaching and bonded with the total-etch adhesive system; and (BTSE), treated with 35% hydrogen peroxide and bonded with the self-etching adhesive system. Shear bond strength was measured using a universal testing machine (EMIC). The adhesive remnant index (ARI) score was verified. The data were analysed using a two-way ANOVA and Tukey test ($p < 0.05$).

Results – Significant differences were found, and the self-etching adhesive groups presented the highest bond strength values (CTSE = 11.55 ± 2.85 MPa; BTSE = 14.14 ± 2.23 MPa). The ARI scores revealed significant differences among the groups; the greater amount of remaining adhesive was observed in the CTSE group, and the lowest scores were observed in the BT group.

Conclusions – The use of the self-etching adhesive system, even after intracoronary bleaching, presented satisfactory adhesive strength for the bonding of brackets.

Key words: dental bonding; orthodontic brackets; shear strength; teeth bleaching

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Introduction

Tooth bleaching is a widely used procedure that has a significant effect on orthodontic practices. For the treatment of teeth discoloured due to endodontic problems, internal tooth bleaching becomes a viable option for re-establishing the harmony of the smile (1). Whitening systems with different concentrations and applications of reagents have been used (2), and studies (3–5) have been performed to evaluate the association between external tooth bleaching and the capacity of brackets to bond to the tooth structure. When used for *external* tooth bleaching, 35–38% hydrogen peroxide solutions can reduce the bond strength of orthodontic accessories when bonded within the first 24 h (4, 5) following bleaching, which may occur due to residual peroxide or oxygen on the tooth structure (6). Therefore, it is recommended that the placement of fixed orthodontic appliances be postponed until bleaching treatments have been completed (2). Based on these concepts, studies (7–9) have been conducted to evaluate the influence of *internal* tooth bleaching on enamel adhesion; however, such studies remain rare and present contradictory results. Some studies have shown a reduction in bond strength (7, 8), whereas others (9) have suggested that the bonding procedure can be performed immediately after internal bleaching without loss of adhesion.

Adhesives used in orthodontic practices can be categorised depending on the surface treatment of the tooth: total acid etch (etch and rinse) using phosphoric acid at 30–37.5% and self-etching primer, which consists of a combination of acidic compounds and monomers in a single product (10). There is no consensus regarding the superiority of total-etch systems compared with self-etching systems for bonding orthodontic brackets (11, 12). Although bond strength values are typically higher for self-etching systems (13), previous studies have also shown that self-etching systems are inferior to the traditional total-etch systems (14). Thus, few investigations have evaluated the influence of internal tooth bleaching on conventional adhesive systems

(7, 8), and no studies have assessed the influence on self-etching systems for the bonding of orthodontic brackets.

This study aimed to evaluate the bond strength of orthodontic brackets bonded with conventional and self-etching adhesive systems immediately after internal tooth bleaching. The null hypotheses tested were that there is no significant difference in the bond strength of brackets placed after internal bleaching and that there is no significant difference between brackets bonded with total-etch and self-etching adhesive systems.

Materials and methods

The study was approved by the Committee on Ethics and Animal n°.06/2011. Sixty freshly extracted bovine incisors were used as a proxy for human teeth. The criteria for tooth selection included dental enamel without cracks/fractures and without restorations or previous applications of chemicals such as thymol, hydrogen peroxide, alcohol, or formalin. The sample size was calculated by adopting a 5% significance level, a test power of 80%, and an effect size of 1.1 with 14 samples per group.

The teeth were cleaned and washed, and the bovine roots were sectioned at the cervical third. The pulp was extirpated, and the thickness of the dentine/enamel on the labial aspect was measured using a thickness gauge. A reduction on the internal surface of the pulp chamber was performed to standardise the thickness at 3 mm.

The samples were randomly divided into four groups of 15 teeth each (Table 1). The samples in the control groups CT and CTSE were stored

Table 1. Experimental groups and treatments

Group	Treatment
CT	Control + Transbond XT
CTSE	Control + Transbond Self-Etching
BT	Intracoronary Bleaching + Transbond XT
BTSE	Intracoronary Bleaching + Transbond Self-Etching

in artificial saliva until the brackets were bonded, and the samples in the bleached groups BT and BTSE were subjected to intracoronary whitening treatments using 35% hydrogen peroxide (Table 2).

The Whiteness HP Maxx was mixed, inserted into the pulp chamber and left for 15 min, with occasional stirring to release any oxygen bubbles that formed and to improve the contact of the gel with the tooth, according to the manufacturer's instructions. The gel was aspirated using an endodontic suction tip, and the pulp chamber was rinsed and dried to receive a fresh layer of whitening agent. Six applications were used to simulate the clinical treatment performed in two sessions with three applications of whitening gel per session.

After the intracoronary bleaching treatment, the pulp chambers were obliterated using soft wax, and the samples were embedded in acrylic resin within polyvinyl (PVC) cylinders and positioned such that the most central and prominent portion of the buccal enamel surface remained parallel to the shear test device.

The Transbond™ XT (3M) conventional adhesive system was used to bond the orthodontic brackets for the CT and BT groups, and the Transbond™ Plus Self Etching Primer (3M) system was used for the CTSE and BTSE groups (Table 1).

Prior to bonding of the brackets, prophylaxis of the buccal surface was performed using a

Robson brush and extra-fine water-based pumice paste without fluoride for 10 s. For the CT and BT groups, 37% phosphoric acid was applied for 30 s. After abundant rinsing and air drying, the total-etch Transbond™ XT (3M) system was applied in gentle strokes using a microbrush followed by light air jet and light-curing for 20 s. For the samples in groups CTSE and BTSE, two layers of the adhesive Transbond™ Plus Self Etching Primer (3M) were applied using microbrush strokes for 3 to 5 s and dried with air jets to leave a fine film of adhesive on the enamel surface.

Following application of the adhesive systems, stainless steel brackets for the upper central incisors with a base mesh of 1.8 mm in height and 4.4 mm in width (Edgewise Standard 0.022" × 0.028" Kirium – Abzil, Sao José do Rio Preto, SP, Brazil – Batch: 2011010044) were positioned on the tooth surface aided by orthodontic tweezers (Morelli, Sorocaba, SP, Brazil). The brackets were bonded with Transbond™ XT resin and were light-cured for 20 s on each surface (distal and mesial) after removing the excess resin with a dental probe (Duflex n° 5; SS White, Rio de Janeiro, RJ, Brazil).

The curing was performed using an Optilight Plus light device (Gnatus, Ribeirao Preto, SP, Brazil) with a light intensity of 800 mW/cm² measured using a radiometer. The procedures were performed by a single operator who was appropriately trained and calibrated.

Table 2. Materials and characteristics

Material (manufacturer)	Composition	Batch
Whiteness HP Max 35% (FGM Dental Products, Joinville, Brazil)	35% Hydrogen peroxide	230211
Condac 37 (FGM Dental Products)	36% Phosphoric acid gel, pH <1	191110
Transbond TM XT (3M Unitek, Monrovia, CA, USA)	Orthodontic adhesive system primer: triethyleneglycol-dimethacrylate, bis-MA adhesive: paste silica, bis-GMA, silane, <i>N</i> -dimethylbenzocaine, hexafluorophosphate)	1034300166
Transbond TM Plus Self Etching Primer (3M Unitek)	Orthodontic adhesive system with acid and primer combined (propenoic acid, methyl bisphosphonic ester, HEMA phosphate, camphorquinone, hexafluorotitanate)	1109600297

The specimens were immersed in artificial saliva and stored in an incubator at $37 \pm 1^\circ\text{C}$. After 24 h, the shear bond strength test was performed using a Universal Testing Machine EMIC DL 2000 (EMIC, Sao Jose dos Pinhais, PR, Brazil). A 50-kg load was applied parallel to the buccal surface of the enamel near the enamel/adhesive junction at a speed of 0.5 mm/min until a fracture occurred. The force needed to remove the brackets was measured in Newtons (N), and the shear strength was measured in MPa.

The samples were analysed using a stereoscopic lens (Kozo Optical and Electronical Instrumental, Nanjing-Jiangsu, China) with $10\times$ magnification to assess the adhesive remnant index (ARI). The measurement was performed according to scores that varied from 0 to 3 (15): 0- no remnants of resin on the enamel; 1- less than half of the resin adhered to the enamel; 2- more than half of the resin adhered to the enamel; and 3- all of the resin adhered to the enamel with imprints of the bracket mesh.

Statistical analysis

The Kolmogorov–Smirnov test was used to confirm that the data followed a normal distribution, and the Bartlett test illustrated the homoscedasticity (homogeneity of variances) between the groups.

The data were analysed using Statistica Version 5.1 (StatSoft Inc., Tulsa, OK, USA) using two-way ANOVA and Tukey *post hoc* tests. For the ARI, the Kruskal–Wallis and Dunn tests for multiple comparisons were applied at a significance level of 5% ($p < 0.05$).

Results

The descriptive statistics for the debonding strengths of different combinations of intracoronary treatment and adhesive systems are presented in Table 3.

The two-way ANOVA indicated that the interaction effect (bleaching \times adhesive) was statistically significant ($F = 23.25$, $p < 0.001$). Tukey test (5%) demonstrated that lower values of bond

strength were found in the group treated with the total-etching adhesive system after the intracoronary bleaching treatment. The BTSE and CTSE groups showed the highest mean bond strength values and were not significantly different from one another (Table 3).

Analysis of the adhesive remnant index

The Kruskal–Wallis and Dunn tests demonstrated statistically significant differences in the failure mode and frequency distribution of scores among the groups ($H = 18.43$; $p < 0.001$) (Table 4).

The highest bond strength values were obtained for the groups in which self-etching adhesive was used (BTSE and CTSE), with predominant scores of 2 and 3 (Table 4). In these groups, the failures predominantly occurred at the resin–bracket interface; however, when the failure occurred at the enamel–resin interface ($n = 3$), there were cohesive fractures in the enamel in 20% of the samples in the BTSE group.

Following the total-enamel etch procedure (CT and CTSE groups), the most abundant scores were 0 and 1, and a cohesive enamel fracture was observed in one sample from the CT group (Fig. 1).

Discussion

The number of patients who receive tooth bleaching in dental offices has increased because of the increasing number of orthodontic treatments in adults and a greater emphasis on aesthetics. In this study, the influence of internal

Table 3. The highest and lowest shear strength values (MPa), mean, standard deviation (SD) and Tukey test results for the test groups are shown

Experimental group	N	Mean (SD)	Highest	Lowest	Tukey*
CT	15	11.06 (3.63)	17.07	4.83	A
CTSE	15	11.55 (2.85)	15.68	9.01	AC
BT	15	6.43 (2.70)	10.86	1.14	B
BTSE	15	14.14 (2.23)	18.42	10.76	C

*Groups with different letters are significantly different from each other ($p < 0.05$).

Table 4. Distribution of ARI scores [n (%)] and significant differences between the test groups

Group (n = 15)	ARI score				Mean score (Median)	Dunn*
	0 (%)	1 (%)	2 (%)	3 (%)		
CT	3 (20.0)	7 (46.7)	2 (13.3)	3 (20.0)	1.33 (1)	A
CTSE	1 (6.7)	0 (0)	5 (33.3)	9 (60.0)	2.47 (3)	B
BT	6 (40.0)	7 (46.7)	1 (6.7)	1 (6.7)	0.80 (1)	A
BTSE	3 (20.0)	0 (0)	7 (46.7)	5 (33.3)	1.93 (2)	B

*Groups with different letters are significantly different from each other ($p < 0.05$).

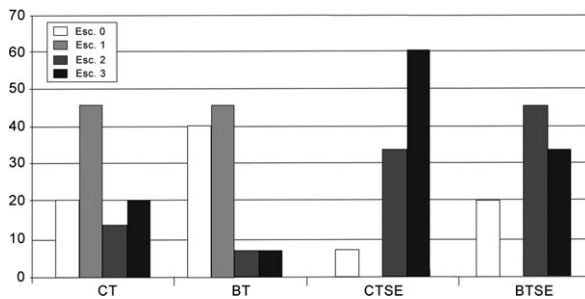


Fig. 1. Distribution of the ARI scores for the four assessment groups.

tooth bleaching treatment on the bond strength of fixed orthodontic brackets after the application of 35% hydrogen peroxide into the pulp chamber was evaluated *in vitro*. The interactions with self-etching and total-etch systems adhesive systems were analysed, and the proposed null hypothesis was partially accepted.

Bovine teeth have been used as a viable alternative to human permanent teeth for bond strength tests in laboratory-based research (16, 17) because they demonstrate micromorphological features similar to human teeth (18).

Hydrogen peroxide at a concentration of 35% was used in this study because it demonstrates a low molecular weight, high enamel/dentine penetration, and the capacity to alter the nature of pigment macromolecules (19, 20). In addition, hydrogen peroxide demonstrates a higher efficacy than does carbamide peroxide (another commonly used whitening agent) at the same concentration. Upon contact with tooth tissue, free radicals are released and penetrate the dentinal tubules (1), which may lead to alterations in the peritubular and intertubular dentine, including increased permeability and reduced elasticity and microhardness of the dentine (20).

Previous studies have suggested that the ideal bond strength of brackets to the enamel should be clinically acceptable and able to resist orthodontic and masticatory forces ranging from 5.9 to 7.8 MPa (21). The maximum bond strength should be less than the cohesive enamel strength, which is approximately 12.75 MPa, to allow the bracket to be removed without enamel lesions (22). In the present study, the mean bond strength values ranged from 6 to 14 MPa, and lesions to the enamel were more frequently found in the groups with higher bond strengths.

There is no evidence in the literature of an association between self-etching adhesive systems and internal tooth bleaching; however, in this study, we observed that the interaction between the application of adhesives immediately after tooth whitening was statistically significant. The type of adhesive system strongly influenced the adhesion because the teeth that were bleached and bonded using a total-etch adhesive system exhibited a lower bond strength compared with teeth bonded using a self-etching adhesive. The results are consistent with studies by Uysal et al., (7, 8) in which the bonding of brackets with a total-etch system immediately after intracoronary bleaching caused a decrease in the bond strength. Possible explanations for the reduced bond strength include the presence of residual oxygen, a decrease in the amount of calcium and phosphate and alterations in the morphology of the hydroxyapatite crystals (3, 20) due to the application of the whitening gel inside the pulp chamber.

Decreases in bond strength can be observed in restorations placed up to 7 days after intracoronary bleaching using sodium perborate, hydrogen

peroxide or carbamide peroxide (23). However, Arcari et al. (9) did not observe a decrease in the adhesive capacity of restorations following internal bleaching, which can be influenced by the type of system that is used. Systems that contain ethanol may minimise the effect of residues left by the whitening agent (24). The self-etching system used in this study contained reactive esters derived from carboxylic or phosphonated acid as reactive components, which form a bifunctional molecule (25). This system allows for a more superficial etching pattern compared with the total-etch system, which can prevent interactions with residual oxygen derived from the whitening agent and might explain the greater adhesion values observed in the CTSE and BTSE groups.

The shear bond strength was greater when the self-etching system was used for the treated and untreated groups, according to the study by Buyuklymaz et al. (13). Due to the high sensitivity of the bonding in the techniques that are used, the steps performed during the procedures will influence the final bond strength results. In the present study, two layers of self-etching agent were applied under friction, which ensured the adhesive efficacy of the system.

In addition to the potential to demineralise and simultaneously promote the imbrication of resin monomers in the tooth substrate, the quantity of acidic monomer, the number of bottles/stages and the initial pH (26) are also important factors in the self-etching systems. The level of demineralisation is related to the pH of the acidic monomers, and monomers with a lower pH will be more aggressive and demonstrate a more defined etching pattern (26). The self-etching system used in this study had a pH of 1, which is considered low among self-etching systems (27); however, the pH of 35% phosphoric acid ranges from 0 to 0.4. Nevertheless, pH is not the only factor that affects the bond strength (26, 27).

The ARI analysis revealed significant differences among the groups. With the application of the self-etching system (CTSE and BTSE), there was a predominance of failures between the resin and the bracket independent of the presence or absence of the bleaching treatment; this finding demonstrated a favourable behaviour of the self-

etching adhesive (11, 13, 14) because studies have shown that it is preferable to remove excess resin from the enamel, thereby avoiding possible unexpected damage to the dental structure. In contrast to the results of this study, other studies (28, 29) have reported failures predominantly at the enamel/resin interface, which is likely attributable to a deficiency in the penetration of the adhesive into the enamel. When using the total-etch system after bleaching treatment (BT), as well as in the control group (CT), failures predominantly occurred at the enamel/resin interface, which has been previously reported (7, 8). After the shear bond test, damage to the tooth surface was observed in the BTSE group, which demonstrated the highest mean bond strength at debonding, and cohesive enamel fractures occurred in 20% of the samples. However, the shear strength may exhibit lower values when evaluated under *in vivo* conditions compared with *in vitro* studies (30, 31).

Based on the obtained results, the self-etching system, when appropriately applied, may represent a viable alternative for clinical use in orthodontics. However, further clinical and laboratory studies are necessary to identify and correlate the aspects related to the clinical application of the self-etching system.

Conclusions

In view of the experimental conditions and the obtained results, it is possible to conclude the following:

- In intracoronally bleached teeth, the use of a self-etching system produced higher bond strengths than did the conventional adhesive. However, in non-bleached teeth, the conventional and self-etching adhesive systems were not significantly different.
- The teeth bonded with the self-etching adhesive system, combined or not combined with intracoronary bleaching, exhibited a predominance of failures between the resin and the bracket. The overall acid conditioning, associated or not associated with intracoronary

bleaching, yielded more frequent local system failures in the enamel/resin interface.

Clinical relevance

The findings of this study suggest that the intracoronary bleaching not affect the adhesive

strength of the self-etching adhesive system for the bonding of metallic brackets.

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