Shear bond strength of orthodontic fluoride releasing color change adhesive: an in vitro study

Resistência ao cisalhamento de um adesivo ortodôntico com liberação de flúor e mudança de cor: um estudo in vitro

Resistencia al cizallamiento del adhesivo de cambio de color liberador de flúor para ortodoncia: un estudio in vitro

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Abstract
Objective: To assess, in vitro, the shear bond strength and the adhesive remnant index (ARI) of brackets bonded with Transbond™ Plus Color Change adhesive. Design: Laboratory study of shear bond strength. Setting: Orthodontic brackets were bonded on bovine teeth and submitted to a mechanical shear test. Methods: Forty-five bovine teeth were randomly divided into three groups for orthodontic bracket bonding with Transbond™ XT adhesive light cured for 10 seconds (group XT10) and Transbond™ Plus Color Change light cured for 5 seconds (CC05 group) and 10 seconds (CC10 group). The specimens were submitted to a mechanical shear test and the enamel was examined under the stereoscopic microscope to determine the ARI. Results: There was no significant difference in shear strength between the groups, considering adhesive type ($p = 0.137$) and polymerization time ($p = 0.958$). According to the ARI scale, 86.7 % of the teeth in CC10 and CC05 groups presented more than half of the adhesive left on them, while in XT10 group 73.3 % of the teeth presented less than half of the adhesive left on them, and this difference was significant. Conclusion: The use of the Transbond™ Plus Color Change adhesive resulted in a non-significant reduction in the shear bond strength and in a greater adhesive/bracket interface failure when compared to Transbond™ XT.
Keywords: Orthodontics; Dental bonding; Fluorine; Shear strength.

Resumo
Objetivo: Avaliar, in vitro, a resistência ao cisalhamento e o índice de adesivo remanescente (IAR) de bráquetes colados com o adesivo Transbond^{TM} Plus Color Change. Desenho: Estudo laboratorial da resistência ao cisalhamento. Metodologia: Bráquetes ortodônticos foram colados em dentes bovinos e submetidos a teste de cisalhamento mecânico. Métodos: Quarenta e cinco dentes bovinos foram divididos aleatoriamente em três grupos para colagem de bráquetes ortodônticos com adesivo Transbond™ XT fotopolimerizado por 10 segundos (grupo XT10) e Transbond^{TM} Plus Color Change fotopolimerizado por 5 segundos (grupo CC05) e 10 segundos (CC10 grupo). As amostras foram submetidas ao teste de cisalhamento mecânico e o esmalte foi examinado ao microscópio estereoscópico para determinação do IAR. Resultados: Não houve diferença significativa na resistência ao cisalhamento entre os grupos, considerando o tipo de adesivo (p = 0,137) e o tempo de polimerização (p = 0,958). De acordo com a escala IAR, 86,7% dos dentes dos grupos CC10 e CC05 apresentaram mais da metade do adesivo remanescente, enquanto no grupo XT10 73,3% dos dentes apresentaram menos da metade do adesivo remanescente, e essa diferença foi significativa. Conclusão**:** O uso do adesivo Transbond^{TM} Plus Color Change resultou em redução não significativa da resistência de união ao cisalhamento e maior falha na interface adesivo/bráquete quando comparado ao Transbond^{TM} XT.
Palavras-chave: Ortodontia; Colagem dentária; Flúor; Resistência ao cisalhamento.

Resumen
Objetivo: Evaluar, in vitro, la resistencia al cizallamiento y el índice de adhesivo remanente (ARI) de brackets cementados con el adhesivo Transbond^{TM} Plus Color Change. Diseño: Estudio de laboratorio de resistencia al cizallamiento. Metodología: Los brackets de ortodoncia se adhirieron a dientes bovinos y se sometieron a prueba de cizallamiento mecánico. Métodos: Cuarenta y cinco dientes bovinos se dividieron aleatoriamente en tres grupos para la cementación de brackets de ortodoncia con adhesivo Transbond™ XT fotopolimerizable durante 10 segundos (grupo XT10) y Transbond™ Plus Color Change fotopolimerizable durante 5 segundos (grupo CC05) y 10 segundos (grupo CC10). Las muestras se sometieron al ensayo de cizallamiento mecánico y se examinó el esmalte al microscopio estereoscópico para determinar el ARI. Resultados: No hubo diferencia significativa en la resistencia al cizallamiento entre los grupos, considerando el tipo de adhesivo (p = 0,137) y el tiempo de polimerización (p = 0,958). Según la escala ARI, el 86,7% de los dientes de los grupos CC10 y CC05 tenían más de la mitad de adhesivo remanente, mientras que en el grupo XT10 el 73,3% de los dientes tenían menos de la mitad de adhesivo remanente, siendo esta diferencia significativa. Conclusión: El uso del adhesivo Transbond^{TM} Plus Color Change resultó en una reducción no significativa en la resistencia de la unión al cizallamiento y una mayor falla en la interfaz del adhesivo/bracket en comparación con Transbond^{TM} XT.
Palabras clave: Ortodoncia; Recubrimiento dental adhesivo; Flúor; Resistencia al corte.

1. Introduction

Orthodontic bonding with light-cured adhesives became popular among orthodontists mainly due to its longer working time for appliances positioning compared to self-curing adhesives (Bishara et al., 2003; Lamper et al., 2014). The adequate polymerization of the adhesive is essential for the success of the procedure and it is related to the output intensity of the light applied on it. The light emitting diode (LED) light curing units are distinguished by producing high-intensity light (Fleming et al., 2013), in addition to not emitting ultraviolet and infrared radiation, presenting low heat emission (Malkoç et al., 2010).

The improvement of orthodontic adhesives associated with LED curing unit have allowed the reduction of the exposure time to light without compromising bond strength between orthodontic appliances and dental surfaces, hence simplifying bonding procedure. The introduction of hydrophilic adhesive systems in orthodontic practice provided better results regarding bracket/tooth adhesion (Santos et al., 2010; Faltermeier et al., 2007), since bonding under ideal clinical conditions is not often possible and moisture contamination on the surface of the enamel decreases resin penetration (Santos et al., 2010).

The use of fixed appliances increases the risk of white spot lesions adjacent to orthodontic brackets once they facilitate the retention of bacterial plaque (Buschang et al., 2019). These lesions are caused by enamel demineralization due to the action of acids produced by cariogenic bacteria (Faustova et al., 2018; Raji et al., 2014). Thus, an ideal adhesive system must have sufficient bond strength to withstand the stresses exerted on bonded brackets and, at the same time, hinder enamel demineralization.

The Transbond™ Plus Color Change Adhesive (3M Unitek) has hydrophilic property and allows fluoride diffusion (Bhushan et al., 2021), which is responsible for its cariostatic activity (Buschang et al., 2019). Furthermore, this adhesive has in its composition coloring agents that change its initial pink color to translucent after light curing. The pink color allows an efficient removal of adhesive excess during bonding, especially when aesthetic brackets are used, which is related to lower bacterial plaque accumulation during treatment (Maurya et al., 2011; Pseiner et al., 2010).

The aim of this in vitro study was to assess the shear bond strength and the Adhesive Remnant Index of brackets bonded with the Transbond™ Plus Color Change adhesive using LED curing light at different exposure times.

2. Methodology

Forty-five lower incisors were extracted from bovine mandibles and after manual removal of all adhered tissue, they were stored in 0.5 % thymol solution at room temperature for seven days. Bovine teeth were used due to the similarity of their enamel to the enamel of human teeth regarding physical properties and composition (Oesterle et al., 1998).

The buccal surfaces of the crown were inspected with a stereomicroscope (Stemi 2000C - Zeiss, Toronto, Canada) and teeth with evidence of fracture, caries or other irregularities on these surfaces were excluded. The crowns were sectioned from the roots and embedded in self-curing acrylic resin (Meliodent, HerauseKulzer, Hanau, Germany). The buccal surfaces remained free and perpendicular to the long axis of the resin cylinder, forming the specimens. The buccal surfaces were abraded with progressively thinner carbide abrasive paper (granulation 220, 400 and 600) in constant water flow and kept in distilled water at room temperature prior to bonding the brackets.

The specimens were randomly divided into 3 groups, according to Table 1, where stainless steel incisors brackets (Mini Standard Edgewise, American Orthodontics, Sheboygan, USA) with 9.73 mm^2 base area were bonded with Transbond™ Plus Color Change adhesive or with Transbond ™ XT adhesive, the latter used as a control.

Table 1: Description of the groups according to the adhesive and the exposure time.

Group	*Adhesive*	*Exposure time*
XT10	Transbond™ XT	10 seconds
CC10	Transbond™ Plus Color Change	10 seconds
CC05	Transbond™ Plus Color Change	5 seconds

Source: 3M Unitek, Monrovia, USA.

Bracket Bonding Procedure:

Initially, the enamel surfaces of each specimen were polished with pumice (Bimsstein Pulver, Prochimie, Switzerland) for 15 seconds, rinsed with water and dried with an oil and water-free air jet for 10 seconds. Transbond™ Self Etching Primer (3M Unitek, Monrovia, USA) was then applied for 5 seconds, followed by a gentle air-dry.

The orthodontic adhesive (Transbond™ Plus Color Change or Transbond™ XT) was applied to the bases of the brackets which were positioned in the center of the flat area of the buccal surfaces of dental crowns and a force of 300 g was applied for 5 seconds using a spring balance (Correx Tension Gauge, Haag-Streit AG, Koeniz, Switzerland). The excess of adhesive was carefully removed with a dental explorer.

The adhesives were photopolymerized using a LED curing unit (Elipar Freelight 2 LED Curing Light - 3M Unitek, Monrovia, USA) with light intensity of 900 mW / cm^2 and the exposure time was monitored as described in Table 1. The exposure time was equally divided between mesial and distal surfaces of the brackets, except for the group CC LED05, in which only the incisal surface of the brackets was exposed to light.

After bonding, the specimens were stored in distilled water at 37º for 24 hours prior to the mechanical shear test.

Shear Test:

Each specimen was loaded using a universal testing machine (EMIC DL2000, EMIC, São José dos Pinhais, Brazil), with the buccal surface of the enamel parallel to the direction of the applied force. The force was applied through a segment of 0.018"steel wire attached to the test machine and supported on the bracket incisal wings. The machine was calibrated to run at a constant speed of 1 mm/min until debonding occurred. The force values, in Newtons (N), necessary to remove the brackets were recorded and divided by the bracket area, to obtain the shear strength values in megapascal (MPa or N / mm^2).

After bracket debonding, the enamel surfaces were examined under the Stereoscopic microscope (Stemi 2000C - Zeiss, Toronto, Canada) with a 10x magnification, in order to determine the Adhesive Remnant Index (ARI), according to the 4-point scale introduced by Artun and Bergland (1984).

Statistical Analysis:

Student's t-test for paired samples was used to compare the shear bond strength between the groups, determining the effect of the adhesive (XT10 x CC10) and the effect of the polymerization time (CC10 x CC05). The ARI scores were compared using the chi-square test. The level of significance was $\alpha = 0.05$. All statistical analyses were performed using the statistical software SPSS 20.0 (SPSS, Chicago, USA).

3. Results

The mean values of shear bond strength (MPa) for the evaluated groups are shown in Table 2. No significant difference was observed in the bond strength between the groups, neither in the verification of the effect of the adhesive type ($p = 0.137$) nor the effect of the polymerization time ($p = 0.958$).

Table 2: Shear bond strength (MPa) of the groups evaluated.

Group	***Average***	***SD***	***Minimum***	***Maximum***
$XT10^{B}$	14.85	3.42	9.49	22.40
$CC10^{AB}$	13.24	2.16	9.02	16.01
$CC05^{A}$	13.20	2.05	9.81	16.28

There was no significant difference for the comparisons between groups with the same letter, according to Student's t-test. Source: Authors.

The distribution of the ARI scores for each group and the result of the chi-square test comparing the groups are shown in Table 3. The statistical test revealed a significant difference between the XT10 group and the CC10 and CC05 groups ($p <0.001$).

Table 3: Distribution of the Adhesive Remnant Index (ARI).

Group*	***ARI (%)***			
	0	*1*	*2*	*3*
XT10[A]	1 (6.7)	11 (73.3)	2 (13.3)	1 (6.7)
CC10[B]	0 (0.0)	2 (13.3)	13 (86.7)	0 (0.0)
CC05[B]	0 (0.0)	2 (13.3)	13 (86.7)	0 (0.0)

0: no adhesive left on the tooth. 1: less than half of the adhesive left on the tooth. 2: more than half of the adhesive remained on the tooth. 3: all adhesive left on the tooth with an impression of the bracket base. *Groups with the same letter did not present significant difference according to the chi-square test. Source: Authors.

In the CC10 and CC05 groups, more than 85 % of enamel surfaces contained more than half of the adhesive remaining on the tooth after debonding (index 2), indicating greater adhesive/bracket interface failure when Transbond™ Plus Color Change was used. In the XT10 group, approximately 73.3 % of the group remained with less than half the adhesive on the enamel surface (index 1), which indicates a higher prevalence of failures in the enamel/adhesive interface when Transbond™ XT was used.

4. Discussion

The development of new technologies related to bonding procedures in Orthodontics aims at achieving an adequate level of debonding resistance of the orthodontic devices, using a minimal clinical chair time. In order to test the efficacy of light curing units and orthodontic adhesives, several studies have used the shear bond strength test to simulate in vitro brackets detachment (Santos et al., 2010; Maurya et al., 2011; Pseiner et al., 2010; Tuürkkahraman & Küçükesmen, 2005).

One of the controversial issues among the studies that perform the shear strength test is the way the force is applied to the specimens. In this study, as has already been reported in the literature (Imani et al., 2018; Fox et al., 1994; Mojtahedzadeh et al., 2006), a steel wire adapted to the bracket incisal wings was used, which allowed a better adaptation to the bracket when compared to the use of the blade placed at the enamel / bracket interface. The use of the blade may cause the force to be applied to the adhesive rather than to the bracket when the enamel/ bracket adaptation is not perfect or there is some adhesive remnant around the bracket (Imani et al., 2016; Foz et al., 1994; Mojtahedzadeh et al., 2016). In addition, one of the main causes of brackets detachment is the resulting forces from masticatory function (Imani et al., 2016; Mojtahedzadeh et al., 2016), when occlusal forces reach the brackets occlusal/incisal wings, similar to the device used to apply the forces in this *in vitro* study.

The aim of this study was to evaluate the shear bond strength of Transbond™ Plus Color Change adhesive due to its chromatic, hydrophilic and fluoride release properties, which facilitates the removal of the excess adhesive around the brackets, reducing the mechanical retention of biofilm and also preventing decalcification and the formation of white spot lesions on tooth enamel[19]. These lesions may compromise the final aesthetic result after the fixed orthodontic appliance removal (Sonesson et al., 2020; Khoroushi & Kachuie, 2017). The protective effects obtained through orthodontic adhesives with fluoride release capacity were confirmed by *in* vivo (Pascotto et al. 2004) and *in* vitro (Raji et al., 2014; Nascimento at al., 2016; Kobayashi et al., 2021) studies. The fluoride ions release also allows remineralization of the enamel attacked by phosphoric acid by the formation of fluorapatite (Kimura et al., 2004).

In the present study, Transbond™ Plus Color Change presented lower values of shear strength than Transbond™ XT ($p = 0.137$), but this difference was not significant, corroborating the results reported in the literature (Santos et al., 2005; Maurya et al., 2011; Pseiner et al., 2010, Devi et al., 2025). All groups presented clinically acceptable bond strength values, which are accepted as being greater than 8 MPa (Türkkahraman et al., 2010). The most commonly tested among all orthodontic adhesives available, Transbond™ XT (3M Unitek) (Maurya et al., 2011; Pseiner et al., 2010; Manfred et al., 2013), was used as a control, similar to previous studies (Santos et al., 2010; Faustova et al., 2018; Maurya et al., 2011; Pseiner et al., 2010; Türkkahraman et al., 2010; Manfred et al., 2013).

Similarly, the study by Maurya et al. (2011) did not find a statistically significant difference in the shear bond strength of brackets bonded with Transbond™ XT and Transbond™ Plus Color Change, either with prior acid conditioning or with self-etching primer. This corroborates the methodology of the present study, in which this primer was used. This new bonding systems use a combination of etching and priming agents into a single primer solution that etches and primers simultaneously the tooth tissues. Active ingredient in self-etching primer is methacrylated phosphoric acid ester. The phosphate group on the methacrylated phosphoric acid ester dissolves the calcium and removes it from the hydroxyapatite. The calcium then forms a complex with the phosphate group and gets incorporated into the network when the primer polymerizes. Continuous rubbing of primer on the tooth surface ensures an uninterrupted flow of fresh primer. Etching and monomer penetration to the exposed enamel rods occur simultaneously. In this manner, the depth of etch is identical to that of the primer penetration (Yadala et al., 2015).

The adhesive remnant index showed a greater amount of adhesive remaining on the bracket surface in most of the specimens (73.3 %) in the group that used Transbond™ XT, which, in clinical practice, reduces clinical chair time because it facilitates adhesive removal and polishing procedures after orthodontic bracket debonding. In the groups that used Transbond™ Plus Color Change, more than 85 % of the specimens had more than half of the adhesive adhered to the tooth, showing lower adhesion capacity of this adhesive to the base of the bracket.

The comparison between the two light curing exposure times, 5 and 10 seconds, applied to Transbond™ Plus Color Change adhesive showed no significant difference in shear bond strength. This has also been shown in previous studies (Mavropoulos et al., 2005; Gupta and Shrestha, 2018), in which the use of a 50 % shorter time implied an average reduction of 0.5 % in shear strength, which is sufficient to withstand orthodontic and masticatory forces (Türkkahraman et al., 2010; Gupta and Shrestha, 2018).

5. Conclusions

The use of Transbond™ Plus Color Change adhesive light cured with LED units for orthodontic bracket bonding resulted in a non-significant reduction of shear bond strength when compared to Transbond™ XT. The IRA indicated a higher prevalence of failure in the adhesive/bracket interface when Transbond™ Plus Color Change was used, with a greater amount of adhesive remaining in the enamel after debonding.

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