Influence of Tooth Bleaching on the Bonding Strength of Silorane-Based Restorative System

Influencia del Aclaramiento Dental en la Fuerza de Adhesion del Sistema Restaurativo a Base de Silorano

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ABSTRACT: Bleaching procedures can interfere on adhesive bonding of methacrylate-based restorative materials. After introduction of silorane-based composites, little information is available about this interaction. The objective is evaluate the influence of bleaching on enamel shear bond strength of silorane-based restorative system. Forty sound incisive bovine teeth were randomly divided into 04 groups (n= 10), being: G1 (control) – not bleached + Clearfil SE Bond (CSEB)/Filtek Z250 (Z250), G2 (control) – not bleached + Silorane Adhesive (SA)/Filtek P90 (P90), G3 – bleached + CSEB/Z250; G4 – bleached + SA/P90. The teeth were sectioned, had crowns included in a PVC rings, and the enamel surfaces gridded and polished with silicon carbide abrasive papers. G1 and G2 were stored in artificial saliva (37 °C), daily renewed, for a period of 04 days. G3 and G4 were exposed to 02 sessions of 38% hydrogen peroxide (38HP), with 03 applications of 15 min in each session. Composite resin cylinders were built up on the enamel surfaces. Bonding procedures were performed according the manufacturer's instructions. After storage in distilled water for 24 h (37 °C), the samples were submitted to shear bond test. Data were analyzed by two-way ANOVA and t (LSD) tests (5%). Intra group comparison showed significant difference for Z250 after bleaching, but not for P90. Shear strength was significant lower for P90, despite bleaching procedures. In conclusion, 38HP bleaching had no influence on the enamel bond strength of P90, even though presenting lower bond strength than Z250.

KEY WORDS: tooth bleaching, composite resins, shear strength.

INTRODUCTION

Tooth bleaching is one of the most requested dental treatments by patients to improve the appearance of their smile. However, neither composites nor porcelain show significant color changes after bleaching treatments, and if restorations of this nature are present in aesthetically critical areas, they will need to be replaced (Lima et al., 2011; Swift & Perdigão, 1998).

Several studies have shown that there is a decrease in bonding strength to bleached enamel/dentin when adhesive restorations are performed directly after different bleaching techniques (Amaral et al., 2008; Nour El-din et al., 2006). This fact can be attributed to morphological and/or chemical changes in substrates, or even to the presence of residual

oxygen in the inner dental tissues, which interferes with the polymerization reaction in adhesive systems and composite resins by inhibiting the generation of free radicals (Burgess & Cakir, 2010; Weinmann *et al.*, 2005). However, it should be emphasized that this information is derived from the use of methacrylate-based resin materials (Nour El-din *et al.*; Hussain & Wang, 2010).

In this context, a new composite based on silorane with low polymerization shrinkage was introduced on the dental market, comprised of different monomers of conventional methacrylate-based resins and a unique adhesive system. The formation of polymers through the opening of silorane rings

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decreases the shrinkage of this material, reducing the possibility of crack formation and microleakage at the tooth/restoration interface (Ilie & Hickel, 2006; Weinmann *et al.*). However, the interference of bleaching treatments with regard to the bonding strength to dental substrates in this category of materials is still largely unknown (Lima *et al.*).

Thus, the aim of this study was to evaluate the influence of prior tooth bleaching, as performed by an in-office technique, on the adhesive bonding strength of silorane and methacrylate-based restorative systems to enamel.

MATERIAL AND METHOD

This study was approved by the Ethics Committee on Research with Experimental Animals of the Federal University of Pará (CEPAE/UFPA) (protocol number: 096/12). Forty sound bovine incisors were rinsed in tap water and stored in a 0.1% thymol solution for 1 week. After disinfection, they underwent prophylactic treatment with pumice paste, and were examined under a stereo magnifier (10X) to ensure that the specimens were free from cracks or defects in the enamel.

Next, the teeth were sectioned at cementumenamel junction (CEJ), and had their remaining crowns mounted in PVC rings with chemically activated acrylic resin. After, the buccal surfaces of the enamel were gridded and polished with silicon carbide abrasive papers (400 and 600) under refrigeration.

The materials used in this study are described in Table I, including the brand name (manufacturer), classification, composition, and batch.

The total sample was randomly divided into 4 groups (n= 10), according to bleaching treatment performance and the adhesive system / composite resinused (Table II).

Control groups (1 and 2) were kept in artificial saliva at 37 °C for a period of 4 days, daily renewed, and were not exposed to any bleaching treatment.

Groups 3 and 4 were subjected to a bleaching agent based on 38% hydrogen peroxide, Opalescence Boost PF (Ultradent, South Jordan, UT, USA), applied to the buccal surfaces in a layer approximately 1 mm in thickness. The gel was left on the substrate for a period of 15 min, after which it was removed with gauze. The application procedure was repeated 2 more times, comprising 1 bleaching session. After 3 days, another bleaching session

Table I. Brand name (manufacturer), classification, composition and batch numbers of materials used in the study.

Brand name (manufacturer)	Classification	Composition	Batch numbers	
Clearfil SE Bond (Kuraray)	02 steps self- etching adhesive system	Primer: MDP, HEMA Camphoroquinone hydrophylic, dimethacrylate, N.N Dietanol ptoluidin and water. Bond: MDP, Bis-GMA, HEMA, hydrophobic camphoroquinone dimethacrylate N.N-Dietanol p-toluidine, Sílanized silica.	201949	
Silorane Adhesive (3M Espe)	02 steps self- etching adhesive system	Primer: Phosphated methacrilate, Vitrebond copolymer, BisGMA, HEMA, water, ethanol and silinized silica particles. Bond: Hydrophobic dimetacrylate, phosphated methacrylate, TEGDMA and silanized silica particles.	1202700211 1221900749	
Filtek Z250 (3M Espe)	Microhybrid composite resin	Aluminum oxide, silica, zirconium oxide, Bis- GMA/UDMA e Bis-EMA/TEGDMA	1130100098	
Filtek P90 (3M Espe)	Low shrinkage microhybrid composite resin	Silica/Silane, yttrium trifluoride bis-3, 4-epoxyciclohexil-etil-fenil-methilsilane, 3,4-epoxicyclohexilciclopolimethilsiloxane	1218500237	
Opalescence Boost PF 38% (Ultradent)	In-office bleaching agent	38% hydrogen peroxide	R7QP8	

Table II. Division of experimental groups.

Group	Bleaching	Adhesive system	Composite resin
1 (control)	No	Clearfil SE Bond	Filtek Z250
2 (control)	No	Silorane Adhesive	Filtek P90
3	Yes (38% Hydrogen peroxide)	Clearfil SE Bond	Filtek Z250
4	Yes (38% Hydrogen peroxide)	Silorane Adhesive	Filtek P90

^{*=} Significative at 5%.

was performed. Between sessions, the specimens were stored in artificial saliva at 37 $^{\circ}$ C. After the second bleaching session, the specimens were kept in artificial saliva for 24 h until the adhesive bonding procedures were performed.

All groups had the area available for adhesion delimited with an adhesive paper disc containing a 3 mm in diameter central hole, placed on the enamel surface. The adhesive systems Clearfil SE Bond (Kuraray, Okayama, Japan) and Silorane Adhesive (3M Espe, Seefeld, Germany), both self-etching primers, were applied according to the manufacturers' instructions.

Composite resin cylinders were prepared over the enamel surfaces by adapting the specimens to a split metallic matrix, which formed a 5 mm in height and 3 mm in diameter central hole. The composites were inserted in two portions and individually photo-activated for 20 s and 40 s for Filtek Z250 and Filtek P90, respectively, using an LED device (Ultra Blue D-2000, DMC, São Carlos, SP, Brazil) with a light intensity of 900±10 mW/cm², as measured by a radiometer.

Immediately after, the specimens were stored in distilled water at 37 °C for 24 h until the performance of the shear test, which was conducted with the use of a metallic matrix ribbon (5 mm) and at a cross-head speed of 0.5 mm/min. Once the mechanical test was completed, the specimens were placed under a stereo magnifier (40X) for observation of fracture patterns.

After compiling the data, an analysis of variance (two-way ANOVA) and t (LSD) test were performed at a 5% significance level.

RESULTS

The mean shear strength values and standard deviations for each group are shown in Table III. Through an analysis of variance (two-way ANOVA), a significant difference was observed among the groups depending on if they were bleached or unbleached (Table IV).

There was a significant reduction in the adhesive bonding strength to enamel adhesive for Filtek Z250 composite resin after tooth bleaching, but the same was not observed with the low-shrinkage Filtek P90 composite (Table V), according to t (LSD) test (5%).

By comparing the 2 composites tested, a difference in adhesive bonding strength to enamel was observed at both time assessment points (before and after bleaching), with Filtek Z250 composite resin showing significantly higher values (Table VI) according to t (LSD) test (5%).

Regarding fracture patterns, 36 specimens (90%) were classified as adhesive, and 4 (10%) as cohesive in composite resin, observed in groups 2, 3, and 4.

Table III. Mean and standard deviation of shear bond strengths of experimental groups.

Group	Bleaching	Adhesive System	Composite Resin	Mean (MPa)	Standard Deviatior (MPa)
G1	No	Clearfil SE Bond	Filtek Z250	4.29	±1.71
G2	No	Silorane Adhesive	Filtek P90	0.60	±0.18
G3	Yes	Clearfil SE Bond	Filtek Z250	2.06	±1.40
G4	Yes	Silorane Adhesive	Filtek P90	0.45	±0.14

Table IV. Two-way Analysis of Variance (ANOVA) for shear bond strengths (MPa).

Treatment	Df	Sum of Squares	Mean Square	F	р
Bleaching	3	95.1015	31.700	25.5323	<0.0001*
Bonding strategy	9	11.136	1.237	0.9966	0.5334
Error	27	33.523	1.242		

^{*=} Significative at 5%.

Table V. Shear bond strength (mean/MPa) according to the bleaching treatment.

Adhaniya System / Composite Bosin	Bleaching Treatment			
Adhesive System / Composite Resin	No Bleaching	Bleaching	τ	р
Clearfil SE Bond / Filtek Z250	4.29	2.06	4.471	<0.001*
Silorane Adhesive / Filte k P90	0.60	0.45	0.297	ns

^{*=} Significative at 5%; ns= No significant.

Table VI. Mean of shear bond strength (MPa) according to composite resins.

Bleaching Treatment	Adhesive System / Composite Resin			n
2 road mily 1 road mont	Clearfil SE Bond / Filtek Z250	Silorane Adhesive / Filtek P90	- •	P
No bleaching	4.29	0.60	7.403	<0.001*
Bleaching	2.06	0.45	3.229	0.0032*

^{*=} Significative at 5%.

DISCUSSION

The possible effects of bleaching treatments on bonding procedures to dental substrates have been the subject of constant research. These studies suggest that using hydrogen peroxide concentrations between 25% and 35% result in a reduction in the ability of resin materials to adhere to previously bleached substrates, as measured by tensile and shear tests (Attin *et al.*, 2008).

The literature recommends a waiting period between 24 h up to 3 weeks after bleaching to perform bonding procedures (Attin *et al.*, 2008; Barbosa *et al.*, 2008; Cavalli *et al.*, 2001; Da Silva Machado *et al.*, 2010; Perdigão *et al.*, 1998; Swift & Perdigão; Unlu *et al.*, 2008; van der Vyver *et al.*, 1997). The main reasons for bonding impairment include a significant reduction in calcium and phosphate levels and morphological changes in the superficial prismatic enamel layer, as well as the possible presence of residual oxygen in the inner part of substrates, which may interfere with the adhesion of composite resins by inhibiting polymerization.

In fact, the subsurface of the enamel organic matrix can be altered by the oxidizing effect of peroxide radicals (Hegedüs *et al.*, 1999; Seghi & Denry, 1992). These alterations may lead to changes in the mechanical properties of the substrate, such as fracture strength (Hegedüs *et al.*). Thus, since fracture toughness is an indicator of the ability of enamel to resist crack propagation, changes in this property would cause a decrease in enamel bonding strength. Likewise, morphological changes would result in impaired adhesive bonding resulting from a loss in mineral content and/or increased porosity (Basting *et al.*). 2001; Perdigão *et al.*).

Such changes in the composition and topography of the enamel seem to be related to factors such as the pH level, the concentration of acidic components, temperature, time, and frequency of exposure to bleaching agents (Basting *et al.*; Price *et al.*, 2000).

Still, bleaching agents act by oxidation, allowing the formation and release of oxygen and hydroxyl radicals. These interact chemically with long chains of pigments and turn them into smaller molecules, causing the visual appearance of teeth whitening (Kawamoto & Tsujimoto, 2004). However, at the end of bleaching therapy, a large amount of residual oxygen may be present in the bleached substrate. The high reactivity of oxygen causes a premature termination of polymer chain formation, compromising the quality of the polymers formed (Rueggeberg & Margeson, 1990), and decreasing the adhesive bonding strength of resin materials to the tooth structure (da Silva *et al.*, 2010; Sasaki *et al.*, 2009).

The reasons listed above may explain the results obtained with the Clearfil SE Bond adhesive system and the Filtek Z250 composite in this study, where performance of a bleaching treatment with high-concentration hydrogen peroxide (38%) significantly reduced the levels of bonding strength to enamel.

On the other hand, while oxidizing compounds used as bleaching agents are able to inhibit free radicalled polymerization of most resin monomers through oxygen release (Tezvergil-Mutluay *et al.*, 2008), the curing reaction by cationic ring opening, which is associated with the presence of oxirane in silorane-based

dental composites, seems to be insensitive to oxygen (Ilie & Hickel, 2009; Shawkat *et al.*, 2009; Tezvergil-Mutluay *et al.*), which could be considered advantageous during adhesion to bleached substrates.

The adhesive system of low shrinkage (silorane) composite resins differs from other 2-step self-etching agents because the self-etching primer requires polymerization (Magni et al., 2008; Navarra et al., 2009), which differentiates it from other methacrylate-based bonding agents (Grégoire et al., 2010; Mine et al., 2010). Navarra et al. showed a higher degree of conversion of silorane adhesive in the hybrid layer than in the Clearfil SE Bond system, which is a fundamental prerequisite for the stability of the adhesive bond.

These explanations may support the results of this study, in which the bonding of the low-shrinkage composite Filtek P90, and its respective adhesive system, were not influenced by prior bleaching with 38% hydrogen peroxide. This finding differs from that of Can–Karabulut and Karabulut (Can-Karabulut & Karabulut, 2011), in which the adhesive strength of low-shrinkage composite resins showed to be sensitive to prior exposure to the same bleaching agent, although in that study peroxide gel was activated by a diode laser during bleaching.

Furthermore, attention should be called to the significantly lower bond strength values obtained with the silorane-based composite Filtek P90 compared to the methacrylate-based composite resin Filtek Z250, regardless of prior bleaching. This result can be attributed, in part, to the chemical differences between the 2 composites.

Guiraldo *et al.* (2010) demonstrated that a difference in light transmission between silorane and methacrylate-based resin composites does exist, with the latter having a significantly higher degree of subsurface polymerization. Moreover, the depth of curing and degree of conversion were statistically lower in the low-shrinkage (silorane) composites than in the conventional (methacrylate) composites (Kusgoz *et al.*, 2011). Thus, as the polymerization of the subsurface is related to adhesive bonding strength (Kim *et al.*, 2006), lower values can be expected with low-shrinkage resins (Guiraldo *et al.*).

The composition of restorative resins also plays a major role in bonding strength to dental substrates. Miyazaki *et al.* (1991) demonstrated that bonding strength is enhanced with the increased filler content of

composites. The composite resin Filtek P90 has a 76% of inorganic content by weight, whereas Filtek Z250 has 82%, which might also explain the results of this study.

Likewise, different photo-activating light sources can influence the strength of the adhesive bond to dental substrates. In a previous study, Khosla *et al.* (2012) showed that the silorane-based composite Filtek P90 had a lower bond strength when photo-activated with an LED-type device compared to activation with a halogen quartz-tungsten lamp.

In the composite resin Filtek P90, one of the photo-initiators is camphorquinone, which is sensitized in the light spectrum of conventional light sources. Still, in the reaction, an electron donor acts in a process of oxidation-reduction, decomposing iodine salts in acidic cations, and initiating the polymerization process by cationic ring opening. Thus, it is assumed that the narrow light spectrum of LED-type devices, as used in this study, may not fully activate these multiple initiators on silorane-based composites (Khosla *et al.*), justifying the lower bonding strength values found in this restorative system.

Still, it has been shown that higher values ??of bonding strength to enamel with the silorane adhesive system are obtained when pre-etching with phosphoric acid is performed, a step which was not carried out in this study. It is believed that the adhesive can penetrate deeper into the intra- and inter-prismatic spaces of the conditioned enamel, improving the performance of the bonding agent and preventing the occurrence of mechanical pretest failures (Duarte et al., 2009), which were common in our research with the Filtek P90 composite.

Finally, one methodological issue deserves to be highlighted: although shear tests represent a commonly used laboratory assessment of the strength of adhesive interfaces (Burke *et al.*, 2008), there has been critiques on its design, as it allows the shearing load to be easily transformed into bending and cleavage forces, generating a non-uniform distribution of tension across the bonding interface and, therefore, compromising the results obtained in terms of a faithful assessment of the strength in the bonding area (DeHoff *et al.*, 1995; Sudsangiam & van Noort, 1991).

These remarks are found with regard to the shear test using a chisel. Thus, considering these limitations, a mechanical test with a metallic ribbon was

chosen, since this would more faithfully allow sliding (shearing) between the enamel surfaces and the composite resin (Braz et al., 2012; Sinhoreti et al., 2001).

Moreover, the low numerical values of bonding strength, as were found in the present study, are typical of shear testing performed with a metal ribbon. If these values are compared with those from similar studies that have used another apparatus during the test (e.g., a chisel), caution should be exercised in drawing conclusions regarding the performance of a given material (Sinhoreti *et al.*).

Because of conflicting data in the literature, it is clear that some doubts still remain regarding the

performance of restorative procedures after the application of bleaching techniques. Therefore, investigation into the occurrence of these laboratory findings in clinical practice is of fundamental importance, given the small amount of information available at this level of assessment.

In conclusion, tooth bleaching significantly reduced the adhesive bonding strength of a methacrylate-based restorative system (Filtek Z250) to enamel, whereas the same was not observed with a silorane-based restorative system (Filtek P90). The adhesive bonding strength to enamel, regardless of performance of a bleaching treatment, was significantly lower with the silorane-based restorative system (Filtek P90).

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RESUMEN: Los procedimientos de blanqueamiento dental pueden interferir en la unión adhesiva de los materiales de restauración a base de metacrilato. Después de la introducción de materiales compuestos a base de silorano, hay poca información disponible acerca de esta interacción. El objetivo fue evaluar la influencia del blanqueamiento en la cizalladura de la fuerza de adhesion sobre esmalte del sistema restaurativo a base de silorano. Cuarenta dientes incisivos bovinos fueron divididos aleatoriamente en 4 grupos (n= 10), siendo: G1 (control) - no aclarado + Clearfil SE Bond (CSEB)/Filtek Z250 (z250), G2 (control) - no aclarado + adhesivo de silorane (AS)/Filtek P90 (P90), G3 - aclarado + CSEB / Z250; g4 - aclarado + AS/P90. Los dientes fueron seccionados, tenían coronas incluidas en anillos de pvc, y las superficies de esmalte fueron molidas y pulidas con papeles abrasivos de silicona de silicio. G1 y G2 fueron almacenados en saliva artificial (37 °C), renovada diariamente, durante un período de 04 días. G3 y G4 se expusieron a 2 sesiones de peróxido de hidrógeno al 38% (38 HP) con 03 aplicaciones de 15 min en cada sesión. Los cilindros de resina compuesta fueron construidos en las superficies de esmalte. Los procedimientos de unión se realizaron según las instrucciones del fabricante. Después del almacenamiento en agua destilada durante 24 h (37 °C), las muestras fueron sometidas al ensayo de resistencia de adhesión al cizallamiento. Los datos fueron analizados por ANOVA de dos vías y prueba t (5%). La comparación intragrupo mostró diferencias significativas para Z250 después del aclaramiento, pero no para P90. La resistencia al corte fue significativamente menor para P90, a pesar de los procedimientos de blanqueamiento. En conclusión, el blanqueamiento con 38HP no tuvo influencia en la resistencia de la unión esmalte de P90, a pesar de presentar una fuerza de unión más baja que Z250.

PALABRAS CLAVE: blanqueamiento dental, resinas compuestas, resistencia a la cizalladura.

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