

EFFECT OF BLEACHING ON SHEAR BOND STRENGTH TO DENTIN OF ETCH-AND-RINSE AND SELF-ETCHING PRIMER ADHESIVES

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ABSTRACT

This study evaluated *in vitro* the influence of enamel bleaching on bond strength to dentin. One hundred and twenty bovine incisors were divided into 12 groups ($n=10$), according to adhesive system: Adper Single Bond 2 (ASB2), Prime & Bond 2.1 (PB), Adhese (AD) and Clearfil SE Bond (SE). The experimental groups were exposed to 10% carbamide peroxide for 4 hours a day over a period of 10 days. After the bleaching, they remained stored in artificial saliva for a further 24 hours or 7 days. The control groups were stored in artificial saliva for 24h. Resin composite (Z250) cylinders were made, and the specimens were stored in distilled water (37°C/24h), until the shear strength test was performed. The results were statistically analyzed using ANOVA 3-way and

Turkey's test ($\alpha < 0.05$). The highest mean value of experimental groups was obtained for ASB2 (7d) and the lowest for PB (24h). Significant differences were observed for bleaching and adhesive system, but not for storage time. Moreover, the interaction between the factors under study was shown to be significant. Statistical differences were observed between the tested adhesives for the different bleaching situations and storage times. It may be concluded that dental enamel bleaching and storage time in artificial saliva interfered with the bond strength to subjacent dentin provided by the Prime & Bond 2.1 and Clearfil SE Bond.

Key Words: Tooth bleaching; Dental enamel; Dentin; Dentin-bonding agents.

EFEITO DO CLAREAMENTO NA RESISTÊNCIA AO CISALHAMENTO À DENTINA DE ADESIVOS CONVENCIONAIS E AUTOCONDICIONATES

RESUMO

Este estudo avaliou *in vitro* a influência do clareamento do esmalte na resistência ao cisalhamento à dentina. Cento e vinte incisivos bovinos foram divididos em 12 grupos ($n=10$), de acordo com o sistema adesivo: Adper Single Bond 2 (ASB2), Prime & Bond 2.1 (PB), Adhese (AD) and Clearfil SE Bond (SE). Os grupos experimentais foram submetidos ao clareamento com peróxido de carbamida a 10% por um período de 10 dias, com exposições diárias por 4 horas. Após o clareamento, permaneceram armazenados em saliva artificial por 24h ou 7 dias. Os grupos controle foram armazenados em saliva artificial por 24h. Amostras de resina composta (Z250) foram confeccionadas e armazenadas em água destilada (37°C/24h), até a realização dos testes. Os resultados foram analisados por meio da ANOVA 3 fatores e teste de Turkey

($\alpha < 0.05$). A maior média para os grupos experimentais foi verificada para o ASB2 (7d) e a menor para PB (24h). Diferenças estatisticamente significante foram observadas para os fatores clareamento e sistema adesivo, porém não foi verificada para o tempo de armazenamento. Em acréscimo, a interação entre as variáveis mostrou-se significante. Diferença estatisticamente significante foi encontrada entre os sistemas adesivos para as diferentes situações de clareamento e tempo de armazenamento. Conclui-se que o clareamento do esmalte dentário e o tempo de armazenamento em saliva artificial interfereu na união à dentina para os adesivos Prime & Bond 2.1 e Clearfil SE Bond.

Palavras-chave: Clareamento de dente; Esmalte dental; Dentina; Adesivos dentinários.

INTRODUCTION

Nightguard vital bleaching is becoming increasingly popular due to its relative ease of use, time-saving and cost-effectiveness¹. The concentration of bleaches applied in whitening procedures varies, depending on the application route and peroxide source².

Current bleaching agents are typically either hydrogen peroxide or carbamide peroxide. There are persistent doubts regarding the clinical use of bleaching agents in their different applications³. The question about the possible interference of bleaching treatments in the bond strength of restorative materials persists because restorations often need to be replaced after bleaching treatment.

Many studies have reported that the adhesive bonding to tooth enamel after bleaching treatment could be damaged if it is performed soon after bleaching therapy⁴⁻⁶. The literature presents highly uniform results in the reduction in bond strength caused by the use of various forms and concentrations of peroxide bleaches⁷⁻⁹. This is attributed to morphological and chemical alterations of the substrate or to the presence of residual oxygen in the enamel, which may be responsible for the inadequate polymerization of the adhesive systems and resin composites used, the release of this oxygen present in the substrate being dependent on the post-bleaching time elapsed^{10,11}.

Bleaching with carbamide peroxide does not involve changes in concentration in oxygen at the

enamel surface, and it is also assumed that dentin could act as an important reservoir of this oxygen, since due to its low molecular weight, the peroxide could easily diffuse into the dentin structure¹². In this context, other studies have shown that enamel bleaching with the different forms of peroxide does not damage the adhesive bond procedures performed afterwards^{5,13}.

Considering all the controversy related to the subject, as well as the frequent need to replace adhesive restorations after bleaching treatments have been performed, the objective of this study was to assess the effect of 10% carbamide peroxide on shear bond strength to dentin of etch-and-rinse, self-etching primer adhesives and exposures to saliva in two post-bleaching periods, as well as to analyze the mode of bond failure.

MATERIALS AND METHODS

One hundred and twenty bovine incisors, previously disinfected in 0.5% chloramine were used. The teeth were randomly divided into twelve groups (n=10), according to whether or not bleaching treatment was performed, type of adhesive system used and storage time until adhesive bonding procedures were performed (Table 1). The groups that were not submitted to bleaching treatment remained stored in artificial saliva (37°C) for the same time as the bleaching group (10 days), followed by an additional period of 24 hours for the adhesive bond procedures to be performed.

Table 1: Division of control and experimental groups, according to bleaching performed, type of adhesive system and storage time.

Bleaching Treatment	Adhesive System and Manufacturer	Storage Time	Group
No (control - G1)	Adper Single Bond (3M ESPE, St. Paul, MN, USA)	24 hours	ASB (24h)
	Prime & Bond 2.1 (Dentsply Caulk, Milford, DE, USA)	24 hours	PB (24h)
	Adhese (Ivoclar/Vivadent, Schaan, Liechtenstein)	24 hours	AD (24h)
	Clearfil SE Bond (Kuraray Medical Inc., Osaka, Japan)	24 hours	SE (24h)
Yes (experimental - G2)	Adper Single Bond (3M ESPE, St. Paul, MN, USA)	24 hours	ASB2 (24h)
		7 days	SB (7d)
	Prime & Bond 2.1 (Dentsply Caulk, Milford, DE, USA)	24 hours	PB (24h)
		7 days	PB (7d)
	Adhese (Ivoclar/Vivadent, Schaan, Liechtenstein)	24 hours	AD (24h)
		7 days	AD (7d)
	Clearfil SE Bond (Kuraray Medical Inc., Osaka, Japan)	24 hours	SE (24h)
		7days	SE (7d)

The specimens in the experimental groups were exposed to 0.1ml of the 10% carbamide peroxide (Magic Bleaching, Vigodent SA Ind. Com., Rio de Janeiro, RJ, Brazil) plus 0.05 ml of artificial saliva for 4 hours daily exposure, for a period of 10 days (manufacturer's recommendation), contained in individual plastic molds made for each tooth with the aid of a vacuum plasticizer appliance Bio-Art Model P3 (Bio-Art - Equipamentos Odontológicos, São Paulo, SP, Brazil). While not in contact with the bleaching agent, the teeth were kept in artificial saliva, renewed daily, at a temperature of 37°C. Before being inserted and when removed from the mold with bleaching agent and artificial saliva, each tooth was washed under running water for 60 s and dried with air jets for 30 s. After the 10 days of bleaching treatment had elapsed, the teeth from the experimental groups were kept in artificial saliva for a further 24 hours or 7 days, after which the adhesive bond treatments were performed.

The enamel surface was ground using 180, 240, 320 and 600-grit silicon carbide paper under running water on a polishing machine (Panambra DP 10, Struers, São Paulo, SP, Brazil) to create a standardized flat surface exposing the superficial dentin substrate. The adhesive systems were used according to the recommendations of the respective manufacturers.

To build the restoration, a round stainless steel mould with a longitudinally cut central hole 5 mm in height and 3 mm in diameter was positioned over the specimens so that the central hole coincided with the delimited area of dentin. Filtek Z250 (3M ESPE, St. Paul, MN, USA) composite resin was inserted in 2 increments, and each was photopolymerized with Ultralux (Dabi Atlante, São Paulo, SP, Brazil) 20s, 500mw/cm².

After the resin composite cylinders had been prepared, the specimens were stored in distilled water (37°) for 24h, and subsequently submitted to shear

bond strength test in a Universal machine (Kratos Equipamentos Industriais LTDA, Taboão da Serra, SP, Brazil). This test was performed with metallic ribbon at a speed of 0.5 mm/min. The metallic ribbon "embraces" the cylinder of composite resin and has its ends fixed to the mechanical claw, which engages the load cell that moves in the vertical direction, up (direction of traction), providing the shear the cylinder of composite resin on the dentin surface. The shear bond strength was calculated and data analyzed using ANOVA with Turkey's test ($\alpha=0.05$).

The specimen fracture patterns were assessed under a stereoscopic microscope at 63X magnification (Nikon SMZ 800, Nikon Corp., Tokyo, Japan), and were classified as follows: Type 1 – Adhesive Fracture (occurring at the bond interface, with adhesive system displacement from the dentinal surface); Type 2 – Cohesive fracture in dentin (dentinal substrate fracture); Type 3 - Cohesive fracture in the resin composite (fracture that occurred in the resin composite structure); and Type 4 – Mixed fracture (fracture with features of adhesive fracture and/or cohesive fracture of adhesive and/or cohesive resin fracture).

RESULTS

The data analysis showed that the highest mean bond strength occurred in experimental group ASB2 (7d), and the lowest in group PB (24h). In the groups submitted to bleaching, the mean bond strength values were higher after storage in artificial saliva for 7 days before the bonding stage.

The comparison of the mean shear bond strength values between the storage times, according to the bleaching and adhesive system combinations showed that there were significant differences between the storage times for each of the bleaching and adhesive system combinations ($p<0.05$) (Table 2).

Table 2: Mean shear bond strength (MPa/SD) with regard to adhesive systems.

Bleaching	Storage Time	ASB2	PB	AD	CF	P value ⁽¹⁾
Control Group	24 hours	4.80±2.65 (a)	3.20±1.84 (a,A)	4.55±2.42 (a)	1.30±0.61 (b,A)	P (1) < 0.001*
Experimental Groups	24 hours	4.17±1.48 (a)	0.85±0.33 (b,B)	3.57±1.64 (a)	1.17±0.64 (b,A)	P (1) < 0.001*
	7 days	5.92±2.16 (a)	3.10±0.82 (b,A)	5.71±2.10 (a)	4.05±2.05 (ab,B)	P (1) = 0.002*
P value		P (1) = 0.202	P (1) < 0.001*	P (1) = 0.088	P (1) < 0.001*	

(*) Significant difference at level of 5.0%; (1) – by F test (ANOVA); Different letters indicate statistically significant difference, by means of the Tukey test. (A) Column; (a) Line.

The comparison of the mean shear bond strength values between samples that were or were not bleached, according to the storage time and adhesive system combinations, for 24 hours storage showed differences only between the samples with bleaching for the adhesive system Prime & Bond, while in the assessment of 7 days of storage showed differences only between the samples with or without bleaching for the adhesive system Clearfil SE Bond ($p < 0.05$). After the shear test stage, the fracture patterns of all the test specimens were assessed. For the groups submitted to bleaching treatment, the fracture patterns of the adhesive type were predominant, once again irrespective of storage time in artificial saliva.

DISCUSSION

Various studies have been conducted with the purpose of assessing the interaction of this bleaching treatment modality on the bonding capacity of restorative materials to dental substrates¹⁴⁻¹⁷. Generally the literature recommends a waiting period after bleaching before performing restorative procedures comprising a time between 24 hours and 3 weeks¹⁸⁻²⁰.

On the other hand, the methodologies of the studies that assess the repercussions of the different forms of dental bleaching on adhesive bonds to substrates vary greatly, which makes it very difficult to compare and settle the results, and contributes to perpetuating the questions regarding the subject³.

Initially, when considering the control group, it was evident that there were statistically significant differences between self-etching Clearfil SE Bond for the other adhesive systems tested. Clearfil SE Bond presents hydrogenionic potential (pH 2.0), and is therefore classified as light or moderate, which comprises self-etching systems with pH between 1.5 and 3.0^{21,22}. This acidity may have been insufficient to promote adequate demineralization of the dentinal substrate, thereby compromising the adhesive bond strength to it.

According to the present study, the applications of the self-etching systems, both Clearfil SE Bond and Adhese were unconditionally made according to the recommendations of the respective manufacturers, who make no type of reference with regard to the active application of the adhesive agents. This could have influenced the bond strength values obtained. The option of working with a noticeably thicker smear layer similar to that obtained with clinical

procedures performed with carbide burs²³, however, does not apply to the above explanation, because the smear layer in this study was standardized using silicon carbide paper.

Therefore, if on the one hand an increase in the thickness and compactness of the smear layer has been directly related to the lower bond strength values provided by self-etching systems, especially self-etching primers^{23,24}, on the other hand, laboratory work with characteristics that approach clinical conditions enables more relevant data to be generated.

Nevertheless, this mean bond strength value obtained for Clearfil SE Bond, should not be considered as the only parameter for reaching a conclusion about the material, since the *self-etching primers* have shown good results, especially when applied to dentin, and have shown some additional clinical benefits, particularly with regard to marginal sealing, in which they have been shown to be superior to the conventional 2-step systems, and comparable to the conventional 3-step systems²⁵. This can also be explained by the presence of the MDP in its composition, which promotes a chemical bond to the dental substrate and could be time-dependent, because after 7 days of storage, its bond strength was higher than that of the control group.

The explanations for these findings in relation to self-etching systems can be compared to those described in the previous paragraphs, reinforcing the fact that the self-etching primer frequently has lower bond strength values when compared to conventional 2 and 3-step systems. Moreover, Prime & Bond 2.1, classified as a conventional 2-step system with acetone as organic solvent, could have its mean bond strength value significantly diminished by the sensitivity of the technique, characteristic of this category of system²⁶.

Therefore, when an acetone-based bonding system comes into contact with humid dentin, there is an abrupt increase in the pressure of the water vapor remaining on the dentinal surface, and if there is not a satisfactorily high quantity, dentin permeability will be rapidly reduced, which may lead to the collapse of the collagen fibers exposed by acid etching, thus making it difficult for the resinous monomers, which do not allow its penetration into the formation of the hybrid layer²⁷.

In this study, the option of using adhesive systems with different solvents is justified by the possibility

of these systems exerting different influences on the bonding process of bleached substrates^{12,16,28}, and could thus contribute to a better understanding of some of the results found.

The presence of alcohol in the primer could be beneficial to the adhesive bond to bleached substrates²⁸. Alcohol could interact with the residual oxygen/peroxides present in the dental structure, minimizing their inhibitory effects on the polymerization of the adhesive system. Furthermore, ethanol is known for its capacity to reduce the surface water content and increase the adhesive bond strength values to enamel¹⁶, and this same reasoning can be transferred to dentin, due to the characteristic humidity of the substrate⁹.

Therefore, the results presented here appear to be in agreement with those of Sung et al.¹⁶, when they demonstrated that the acetone-based adhesive system is more sensitive to the immediate effects of bleaching on the adhesive bond processes. Nevertheless, if the adhesive restorative procedures could be postponed for a longer period of time, the choice of the bond system to be used would become less critical.

However, the significant decrease in bond strength provided by Prime & Bond 2.1 24h after bleaching ended must not be associated only with the previous performance of bleaching therapy, combined with the type of solvent in the bonding agent, since the literature points out the statistical differences between bleached and non-bleached substrates, irrespective of the solvent present in the adhesive system. There is also a reference to acetone-based adhesive systems being able to reverse the adverse effects of the bleaching treatment on the bond strength to dental enamel, thus dispensing the need for any post-treatment waiting time¹⁴.

For a long time it was believed that the reduction in the values of bond strength to bleached enamel were due to the presence of gas bubbles at the resin composite/enamel interface, coming from the oxidation reaction set off by the bleaching agent, or by the existence of residual oxygen/peroxide in the enamel structure, which would contribute to inadequate polymerization of the adhesive systems. Moreover, it was considered that these water-soluble residual composites would be released as a function of time^{14,17,29,30}.

Nonetheless, according to Perdigão et al.¹², in a study using energy dissipation spectrometry (EDS), dental bleaching with carbamide peroxide did not

imply alterations in the relative concentration of oxygen on the enamel surface (at 1 to 5µm depth) and therefore supported the hypothesis that the reduction in adhesive bond strength caused by bleaching might not be associated with the inhibition of polymerization caused by the accumulation of oxygen/peroxide in the enamel structure, but by their accumulation on the dentinal substrate^{9,31}. This information would be in agreement with other surface analysis studies previously conducted, in which bleaching with hydrogen peroxide did not result in oxygen accumulation in the proximity of the bleached enamel³². In addition, this possibility finds support in other studies, in which dental enamel bleaching did not point towards significant reductions in adhesive bond strength to this substrate^{13,15}. In this context, due to the possibility of oxygen/peroxide concentration on the dentinal substrate, which could compromise the adhesive restorative procedures after bleaching, different waiting times have been recommended after the bleaching treatment ends, which could be 24 hours²⁹, 7 days^{9,33,34}, 2 weeks¹⁰ or 3 weeks¹¹. The results of the present study are in agreement with these recommendations, in which the longer post-bleaching storage time of the test specimens may result in a significant increase in the bond strength values, when compared with the adhesive procedure performed 24h after the end of the bleaching treatment. Clearfil SE Bond behaves in this way.

The explanation for this may begin with the capacity of peroxides to diffuse through the dental structure. Due to their low molecular weight (30g/mol), peroxides are able to move freely through the enamel and reach the dentin^{35,36}, which, due to its tubular morphology, functions as an important reservoir of residual oxygen/peroxide¹², which in turn compete to compromise the adequate polymerization of the adhesive system used, and consequently contribute to diminishing the bond strength values.

Additionally, the presence of oxygen/peroxide, with high oxidant power within the dentinal substrate might affect its structure, in view of its lower mineral content and greater percentage of organic matrix⁹, and consequently cause denaturing of the dentinal proteins and morphological alterations, thus compromising the performance of the adhesive restoratives¹².

Accordingly, a waiting time after the bleaching treatment would serve not only to eliminate the

residual oxygen/peroxide present within the dentin, but also to provide the possibility of there being repairs to the morphological and structural alterations to the substrate, since storage in artificial saliva, a remineralizing solution, would favor the recovery of dentinal properties and contribute to a more effective adhesive bond process¹⁰.

Therefore, a careful analysis should be made when considering that bond strength values could have been recovered 7 days after bleaching treatment by a possible remineralizing effect of the artificial saliva. The results found for the experimental groups point towards a greater probability of this reversion of the adverse effects caused by bleaching coming from the longer storage time, or even that the storage time in artificial saliva could have some positive effect in terms of adhesive bond strength values only in the specimens that were submitted to bleaching.

In the present study, a large number of adhesive fractures (type 1) was found for the control groups. Similarly, for the bleached groups, a predominance of adhesive type fractures was observed. The exception occurred for the Adhese adhesive system group submitted to storage for 7 days in artificial saliva, in which the high number of cohesive fractures in dentin should be noted. The transport system of the shear test used in the study was the metal strip, which produces no fulcrum on cylinder of composite resin or cleavage surface, promoting a slip between two interfaces. Therefore, the compression and traction produced perpendicularly to the interface will be lower

than those obtained using chisel or orthodontic wire³⁷. Perhaps the efforts to transfer to the metal strip explain the presence of cohesive fracture in the adhesive layer, probably because the structure that receives the efforts during shear.

As previously mentioned, the presence of peroxides within the dentinal tissue could lead to protein denaturing, morphological alterations and consequently, reduction in the performance of adhesive restorations¹². It could also reduce the microhardness values of the substrate³⁸⁻⁴⁰, thus leading to alterations in the organic and inorganic composition of dentin and compromising its mechanical properties⁴¹.

This being so, and in view of the issues considered, there are still some doubts related to performing restorative procedures after administering bleaching techniques, further research is needed on laboratory findings in the field of clinical practice, considering the small amount of information available at this level of assessment. Further long-term clinical trials are needed to provide additional information on the bleaching effect⁴².

CONCLUSION

Dental bleaching interfered in the adhesive bond strength to dentin provided by the Prime & Bond 2.1 and Clearfil SE Bond system when it was performed 24 hours after the bleaching treatment ended. This was not observed for the other adhesives tested. The longer storage time in artificial saliva significantly increased the values of bond strength to dentin of the group bleached and restored with Clearfil SE Bond.

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