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Effect of Carbamide Peroxide Bleaching Gel Concentration on the Bond Strength of Dental Substrates and Resin Composite

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Clinical Relevance

According it has been demonstrated that dental substrates can recover from mineral loss after 14 days from the last bleaching agent application, there is no scientific information about this effect on the dental substrate-adhesive restoration interface.

SUMMARY

Purpose: This study evaluated the effect of bleaching gel containing 10%, 15% and 20% carbamide peroxide (CP) on the bond strength of dental enamel or dentin and resin composite restorations.

Methods: The buccal surfaces of 12 bovine tooth crowns were conditioned with 37% phosphoric acid, and the adhesive resin Single Bond 2 and the resin composite Filtek Z350 were used to perform the restorations. The blocks were sectioned to obtain bar specimens. Each specimen group (enamel–E, dentin–D) was divided into four subgroups (n=15): S–artificial saliva; 10-10% CP bleaching; 15-15% CP bleaching; 20-20% CP bleaching. CP was applied for six hours daily for two weeks. The specimens were submitted to the σ test in a universal testing machine. The data were analyzed by one-way ANOVA and the Tukey post-hoc test and a correlation analysis (r) was performed.

Results: For Group E, the mean value (± standard-deviation) was 21.86 (± 6.03)a, 18.91 (±

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8.31)ab, 15.43 (± 7.44)b and 10.6 (± 4.94)c for ES, E10, E15 and E20, respectively. For Group D, the σ values were 34.73 (± 4.68)a, 35.12 (± 13.43)a, 29.67 (± 6.84)ab and 24.56 (± 6.54)b for DS, D10, D15 and D20, respectively. A negative correlation between the CP concentration and mean values was observed for both the enamel (r=-0.95) and dentin (r=-0.85) groups.

Conclusion: In the current study, the bond strength of the restoration to enamel and the restoration to dentin were influenced by the application of CP and was dependent on the CP concentration in the bleaching gel.

INTRODUCTION

In the search for an esthetically pleasing smile, dental bleaching is a very useful treatment. Currently, there are two main whitening techniques: in-office (professional) and at-home bleaching. When performed correctly, both techniques are efficient and safe.[1] Previous studies have obtained similar results using different bleaching agent concentrations for at-home and in-office bleaching.[2]

The at-home bleaching technique for teeth with vital pulp has been used for many decades; however, it was only in 1989 that Haywood and Heyman[3] presented the whitening method with 10% carbamide peroxide gel. The at-home technique has the advantages of self-administration by the patient, less in-office time, high safety, low adverse effects and low cost.[4]

The bleaching agent action mechanisms are not completely understood. Nevertheless, it is known that bleaching is based on a complex oxidation reaction in which oxygen-free radicals, due to their low molecular weight, infiltrate through the enamel and dentin substrates. The bleaching agent reacts with the pigment substance that gives it electrons. This reaction results in the opening of pigment carbon rings and converts the rings into clearer intermediate chains, in other words, dental whitening.[3,5-6]

During the whitening process, the agents can cause morphologic alteration of mineralized dental structures.[7-9] In enamel, the alterations are caused by calcium and phosphate loss and the modification that occurs in surface crystals.[10-11] In dentin, organic matrix degradation is also observed and residual oxygen is accumulated. Residual oxygen can make it difficult for the adhesive to impregnate the dentin and inhibit its polymerization. This interferes in the bond strength of adhesive resin to dentin and, consequently, in the biological compatibility of the adhesive system and clinical longevity of the restoration.[11-13]

Enamel bleaching immediately before bonding with adhesive systems damages the bond strength between the materials.[14] Thus, for the bond strength to be re-established, restorative procedures are not recommended anywhere from 24 hours to four weeks after bleaching.[14-15]

The effect of bleaching agents on the bond interface of restorations to dental substrates is controversial. Much of the current research shows marginal sealing alterations[16-17] and a decrease in bond strength after whitening procedures.[18] On the other hand, studies have demonstrated no significant alterations in marginal integrity after bleaching.[19-20] Most available research in the current literature documents performing enamel or dentin whitening before adhesive and restorative procedures.

However, analysis of the influence of bleaching agents on enamel, dentin, adhesive and resin composite on pre-existing restorations that are not indicated for replacement, has been of notable importance in obtaining useful information about tooth-restoration bond degradation. Thus, the main objective of the current research was to conduct an *in vitro* study to evaluate the effects of the application and type (10%, 15% and 20%) of carbamide peroxide bleaching gel on microtensile bond strength between dental substrate (enamel and dentin) and resin composite. The null hypothesis was that the bond strength between dental substrate and resin composite would not be influenced by the bleaching agent application in any of the studied concentrations.

METHODS AND MATERIALS

Fifteen extracted bovine teeth were cleaned and stored in distilled and deionized (H_2O_{dd}) water at -18°C until used. A flexible diamond disc (KG Sorensen, Rio de Janeiro, RJ, Brazil) attached to a handpiece was used to cut the roots from the teeth 2 mm apically to the cement-enamel junction. The teeth were then randomly divided into two groups: Group E (enamel) and Group D (dentin).

In Group E, the buccal enamel was ground and polished with 600 grit silicone carbide paper using a circular polishing machine (DP-10, Panambra, São Paulo, SP, Brazil) under water cooling to obtain a 5 mm^2 area of flat enamel. In Group D, a 5 mm^2 dentin area was exposed following the same protocol utilized for Group E. Measurements were taken to standardize dentin thickness at 2 mm.

The surfaces were etched with 37% phosphoric acid (Scotchbond Etchant, 3M ESPE, St Paul, MN, USA) for 15 seconds in dentin and 30 seconds in enamel and washed with air-water spray for 40 seconds. Excess water was removed with absorbent paper, leaving a moist substrate surface. Subsequently, the adhesive resin Adper Single Bond 2 (3M ESPE) was applied to the treated surfaces according to the manufacturer's instructions and light activated for 10 seconds

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Table 1: *Commercial Names, Composition and Materials' Manufacturers*

Scotchbond Etchand	3M ESPE St Paul, MN, USA	Phosphoric acid 37%.
Adper Single Bond 2	3M ESPE St Paul, MN, USA	Bis-GMA, HEMA, dimethacrylate, methacrylate functional copolymer of polyacrylic and polytaconic acid, water, alcohol, photoinitiator.
Filtek Z350	3M ESPE St Paul, MN, USA	Bis-GMA, UDMA, TEGDMA, Bis-EMA, 20 nm nanosilica and zirconia particles.
Opalescence PF 10 %	Ultradent Products South Jordan, UT, USA	10% carbamide peroxide gel, potassium nitrate and 0.11% fluoride ion.
Opalescence PF 15 %	Ultradent Products South Jordan UT, USA	15% carbamide peroxide gel, potassium nitrate and 0.11% fluoride ion.
Opalescence PF 20 %	Ultradent Products South Jordan UT, USA	20% carbamide peroxide gel, potassium nitrate and 0.11% fluoride ion.

**Bis-GMA, bisphenol A glycidyl methacrylate; HEMA, bisphenol A glycidyl methacrylate; UDMA, urethane dimethacrylate; TEGDMA, triethylene glycol-dimethacrylate; Bis-EMA, Bisphenol A ethoxylate dimethacrylate.*

with a light unit (Curing Light XL 3000; 3M ESPE) with a power density of 600 mW/cm^2.

Nanocomposite resin Filtek Z350 (3M ESPE) was applied to the prepared enamel and dentin surfaces using a divided Teflon mold with an internal dimension of 4 x 4 x 4 mm. Each 2 mm portion was light activated for 40 seconds. Parallel sections measuring approximately 1 mm were made from the mesial to the distal surface and from the cervical to the occlusal surface using a diamond disc attached to a Labcut 1010 (Extec Technologies Inc, Enfield, CT, USA) cutting machine. The sections were made at low speed under water-cooling to prevent stress induction at the bond interface. Commercial brand names, chemical composition and material manufacturers are presented in Table 1.

The specimens were randomly divided into one control subgroup and three experimental subgroups (n=15) according to the carbamide peroxide concentration used in the whitening treatment:

- GS: Control subgroup, specimens stored in artificial saliva
- G10: Bleaching with Opalescence PF 10%
- G15: Bleaching with Opalescence PF 15%
- G20: Bleaching with Opalescence PF 20%

Carbamide peroxide bleaching gels were applied directly to the bond interface of the specimens daily for six hours. At periodic intervals, the specimens were washed under running water to completely remove the bleaching gel and they were then stored in artificial saliva at 37°C.

After a 14-day treatment, the specimens were attached to a microtensile device in a universal testing machine (DL-1000, EMIC, São José dos Pinhais, PR, Brazil) with a 10 kg load cell at a crosshead speed of 1 mm/minute, according to the ISO 11405 Standard. The data, expressed in megapascal (MPa), were submitted to the parametric statistical test (one-way ANOVA) and Tukey post-hoc test at a 5% level of significance. In addition, linear correlation analysis was performed between the mean bond strength values and carbamide peroxide concentration of the bleaching gels (0%, 10%, 15% and 20%) used in each group.

RESULTS

The results of the variance analyses (one-way ANOVA) can be observed in Table 2. The analysis showed a significant difference in bond strength val-

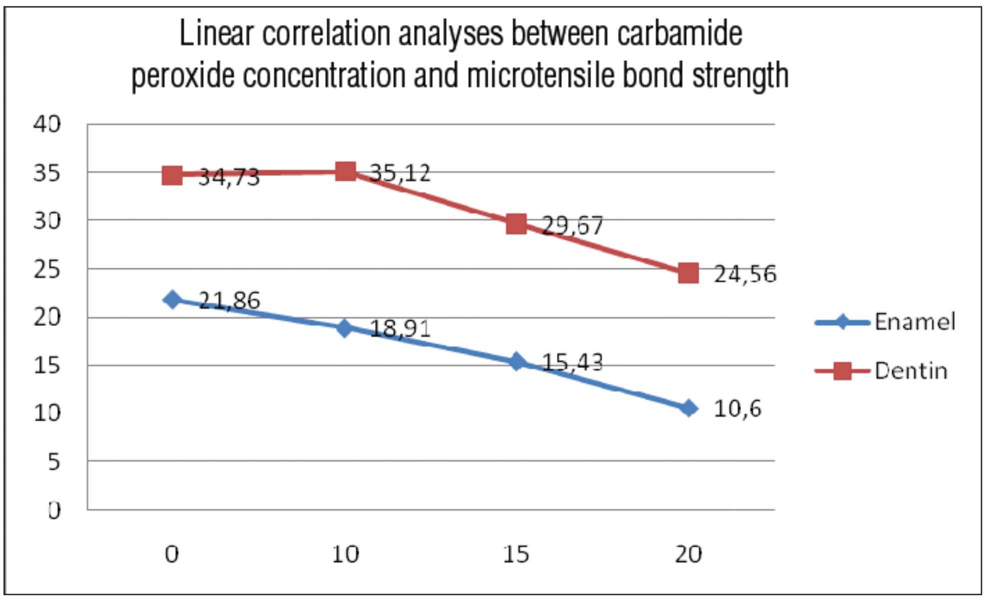

Figure 1. *Correlation analyses between carbamide peroxide concentration (x axis, in %) and microtensile bond strength between dental substrates and restoration (y axis, in MPa).*

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Table 2: *ANOVA Results of Enamel and Dentin Groups*

Groups	Degree of Freedom	F	P
Enamel (E)	3	7.59	0.0001*
Dentin (D)	3	4.90	0.0004*

**Significant differences.*

Table 3: *Mean Bond Strength Values (± standard deviation) and Tukey Post-hoc Results for Enamel Groups*

Subgroup	Mean (± SD)	Homogeneous Groups*		
Control	21.86 (± 6.03)	A		
10%	18.91 (± 8.31)	A	B	
15%	15.43 (± 7.44)		B	
20%	10.60 (± 4.94)			C

**Similar letters mean no significant differences between groups (p≥0.05).*

Table 4: *Mean Bond Strength Values (± standard deviation) and Tukey Post-hoc Results for Dentin Groups*

Subgroup	Mean (± SD)	Homogeneous Groups*	
Control	34.73 (± .68)	A	
10%	35.12 (± 13.43)	A	
15%	29.67 (± 6.84)	A	B
20%	24.56 (± 6.54)		B

**Similar letters mean no significant differences between groups (p≥0.05).*

ues (p<0.005) after 14 days of each whitening treatment for the enamel and dentin groups.

Tukey post-hoc test analyses for the enamel groups are presented in Table 3. The control subgroup is noted to present higher mean bond strength values than the subgroups bleached with 15% and 20% carbamide peroxide.

Table 4 shows the mean bond strength values and differences between the dentin groups as detected by the Tukey test. Similar to the occurrence in the enamel groups, the dentin control subgroup presented higher mean bond strength values than the 20% subgroup.

Linear correlation analyses showed that the higher the carbamide peroxide concentration in the bleaching gel, the lower the bond strength values between the enamel-restoration (r=-0.95) and dentin-restoration (r=-0.85), as observed in Figure 1.

DISCUSSION

In the current study, bovine teeth were used as a substitute for human teeth to evaluate the bond strength between dental substrate and resin composite after at-home dental bleaching. Extracted human teeth are becoming increasingly difficult to obtain, due to recent progress in conservative dental treatment. Histochemical and anatomical studies revealed that all mammalian teeth are essentially similar.[21-22] According to Nakamichi and others,[22] bovine teeth can be used as substitutes for human teeth in the adhesion test, by using enamel and the superficial layer of dentin. Additionally, Saleh and Taymour[23] stated that bovine teeth are a reliable substitute for their human counterparts in bonding studies.

At-home dental bleaching is a conservative treatment of pigmented or stained teeth.[24] The most frequently used substance in the at-home application is carbamide peroxide, which dissociates into hydrogen peroxide and urea. When the pH is more basic, the hydrogen peroxide dissociates and releases oxygen and hydrogen-free radicals, water and peridroxyl. Hydrogen and peridroxyl-free radicals promote the oxidation of pigmented carbon macromolecules, leading to the formation of small, colorless molecules.[5]

The carbamide peroxide (CP) concentration of commercial products varies from 10% to 22%.[24] Matis and others[25] observed no differences in the degree of bleaching achieved in whitening treatments with 10% or 15% carbamide peroxide. However, a much faster bleaching effect was obtained with 15% CP than with 10% CP. Therefore, higher concentrations are used when a faster bleaching result is desired. Considering the action mechanism of the bleaching agent and its capability to diffuse freely through the dental structure, more severe alterations in dental substrates can be observed in treatments with higher concentrations of CP.[26]

The null hypothesis of the current study was rejected for the enamel substrate groups, since the control group presented higher microtensile bond strength values compared with the groups treated with 10%, 15% and 20% CP. It is essential to emphasize that dental enamel is the most mineralized tissue of the human body, with approximately 89% by volume of inorganic elements, 2% organic components and 9% water.[9] The most important inorganic component is hydroxyapatite, formed mainly by phosphate and calcium.

Tames and others[27] verified an increase in the superficial porosity of enamel after treatment with 10% CP for 12 hours of daily application over four weeks. They observed the presence of calcium and phosphate ions, suggesting the formation of basic calcium phosphate, a product generated by hydroxyapatite dissolution. Oliveira and others[28] studied the effects of 10% PC

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used in an at-home whitening technique on dental enamel surface microhardness. The results showed that the bleaching agent produced enamel surface modifications demonstrated by the decrease of microhardness values that started during the first week of CP application. Injury to the enamel surface was intensified after 14 days of treatment. McCraken and Haywood[10] showed that the calcium loss after eight hours of at-home bleaching corresponds to the erosion caused by cola-based soda applied for 2.5 minutes, which is completely reversed by the neutralization and remineralization ability of saliva.

Nevertheless, the current *in vitro* study did not consider the remineralizing action of saliva. In addition to the chemical and morphologic alterations of dental enamel, the absence of the mineralizing power of saliva can explain the bond strength (σ) decrease after the whitening procedure and the strong correlation between the σ values and the CP concentration of the bleaching gel applied.

The null hypothesis of the current study was also rejected for the dentin substrate groups, because the control group presented higher σ values than the group treated with 20% CP. In terms of volume content, dentin presents 45% by volume of mineral content, 30% organic substances, of which the main part is collagen, and 25% water. Several researchers[11,29-30] have verified that the oxidative power of bleaching agents can lead to dentin organic matrix degradation, in addition to increasing dental structure permeability. This effect can be a result of organic components loss during replacement of the carbon groups, hydrocarbon and tertiary amines by oxygen, calcium and phosphorus.[11,29-30]

Moreover, in addition to the enamel and dentin alterations already described, some authors have observed microhardness and surface roughness alterations in resin composites after the bleaching treatment. Bailey and Swift[31] verified some differences in hybrid and microparticulate resin composites after four hours of daily immersion in carbamide peroxide. Turkey and Biskin[32] evaluated the effect of CP bleaching agents on the topography of three esthetic dental materials. These authors demonstrated that, although no differences in surface roughness values were observed for any of the resin composites, the scanning electron microscopy (SEM) analyses showed an enlargement of pre-existent surface porosities after treatment with bleaching agents. This suggests that beaching agents can cause erosion in the composite matrix surface.

As the enamel groups presented a significantly higher decrease in bond strength values after bleaching with 15% and 20% CP than with dentin substrates, it can be affirmed that the enamel-restoration interface was shown to be more sensitive to the action of CP than the restoration bond to dentin substrate. Consequently, the current study demonstrated that the damage caused by the bleaching agent on bond strength is less severe in dentin than in enamel. Moreover, the magnitude of injury depends on the PC concentration used in the whitening procedure. In other words, the higher the PC concentration in the bleaching gel, the lower the tooth-restoration bond strength.

In the current study, the bond strength values between resin composite and enamel are lower than those with dentin. Based on the fact that the dentin of bovine incisors presents larger dentinal tubules and greater porosity of intertubular dentin than human molars,[21] it is easier for the adhesive system to penetrate into bovine dentin than human demineralized dentin. Their longer, thicker tags may induce higher bond strength values in shear or tensile bond strength tests. Corroborating the results of the current study, Lopes and others[33] found bond strength values for enamel substrate lower than those for dentin when bovine substrates were used as substitutes for human teeth.

Carbamide peroxide is supposed to damage the dental substrate bond to resin tags, in other words, the hybrid layer, which is mainly responsible for the mechanisms of adhesion between teeth and resin composites.[34] The authors suggested some protocols to minimize the deleterious effects of bleaching agents on bond interface. Gurgan and others[35] observed that tooth-restoration bond strength values obtained with total-etching adhesives were higher than those obtained with self-etching types. Furthermore, dental surface pretreatment with low-density laser application promotes a better quality of adhesion to resin than to untreated substrate. Thus, the authors suggested using the total-etch technique, laser pretreatment of the dental surface and a waiting time of at least 15 days between the last bleaching session and construction of the direct restoration. Türkün and others[36] demonstrated an increase in shear bond strength values when a resin composite restoration was bonded to an enamel surface pretreated with a solution of 10% sodium ascorbate hydrogel; therefore, this technique could be also indicated for clinical procedures.

Although it has been demonstrated that dental substrates can recover from mineral loss after 14 days from the last bleaching agent application, there is no scientific information about this effect on the dental substrate-adhesive restoration interface.[37-38] Further investigations are needed to clarify and understand this phenomenon.

CONCLUSIONS

The current study showed that carbamide peroxide (CP) bleaching agents could significantly affect the

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microtensile bond strength between the restoration and dental structure. For groups in which the restoration was placed on enamel substrate, the control subgroup presented higher bond strength values when compared with subgroups submitted for bleaching with 15% and 20% CP. For groups with restorations in the dentin substrate, the control showed higher bond strength values compared to the subgroup treated with 20% CP. The damage caused to the restoration-dental structure bond strength by the bleaching agents is augmented with the increase in carbamide peroxide concentration. The resin composite bond to dentin was less sensitive to this adverse effect than the bond to enamel substrate.

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