

## EVALUATING THE BONDING OF TWO ADHESIVE SYSTEMS TO ENAMEL SUBMITTED TO WHITENING DENTIFRICES

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### ABSTRACT

The aim of this study was to evaluate by micro-shear bond strength test, the bond strength of composite resin restoration to enamel submitted to whitening dentifrices. Forty bovine teeth were embedded in polystyrene resin and polished. The specimens were randomly divided into eight groups ( $n=5$ ), according to the dentifrice (carbamide peroxide, hydrogen peroxide and conventional dentifrice) and the adhesive system (Prime & Bond 2.1 and Adper Single Bond 2). Dentifrice was applied for 15 minutes a day, for 21 days. Thirty minutes after the last exposure to dentifrice, the samples were submitted to a bonding procedure with the respective adhesive system. After that, four buttons of resin were bonded in each sample using

transparent cylindrical molds. After 24 hours, the teeth were submitted to the micro-shear bond strength test and subsequent analysis of the fracture mode. Data were submitted to analysis of variance and Fisher's PLSD test ( $\alpha=0.05$ ). The micro-shear bond strength showed no difference between adhesives systems but a significant reduction was found between the control and carbamide groups ( $p=0.0145$ ) and the control and hydrogen groups ( $p=0.0370$ ). The evaluation of the failures modes showed that adhesive failures were predominant. Cohesive failures were predominant in group IV. The use of dentifrice with peroxides can decrease bonding strength in enamel.

**Key words:** dentifrices, tooth bleaching, dental enamel.

## AVALIAÇÃO DA UNIÃO DE DOIS SISTEMAS ADESIVOS AO ESMALTE DENTAL EXPOSTOS A DENTIFRÍCIOS CLAREADORES

### RESUMO

O objetivo deste trabalho foi avaliar, pelo método de microcicatrimento, a resistência adesiva obtida em restaurações de resina composta realizadas sobre o esmalte dental previamente exposto a diferentes dentifícios. Para isso, quarenta dentes bovinos foram incluídos em resina de poliestireno e polidos. Os dentes foram divididos aleatoriamente em 8 grupos ( $n=5$ ) de acordo com o dentífrico utilizado (cremes contendo peróxido de hidrogênio, peróxido de carbamida ou dentífrico convencional) e sistema adesivo (Prime & Bond 2.1 ou Adper single Bond 2). Os dentes foram expostos por 15 minutos por dia durante 21 dias às suspensões dos respectivos cremes dentais. Trinta minutos após a ultima exposição ao dentífrico, os dentes foram restaurados empregando os 2 diferentes sistemas adesivos e resina composta. Após 24 horas os dentes foram

submetidos ao teste de microcicatrimento e posterior análise do padrão de fratura. Os resultados obtidos foram submetidos à análise de variância e teste de Fisher's PLSD ( $\alpha=0.05$ ). Não foi detectada diferença estatística entre os adesivos, porém entre os tratamentos com dentifícios, houve redução estatisticamente significante entre os grupos controle x carbamida ( $p=0.0145$ ) e entre o grupo controle x hidrogênio ( $p=0.0370$ ). Na avaliação do tipo de fratura houve predominância de falhas adesivas, com exceção do grupo IV que apresentou maior fratura coesiva em esmalte. Assim, a utilização de cremes dentais, contendo peróxidos em suas formulações, pode provocar uma redução na resistência de união ao esmalte.

**Palavras chave:** dentífrico, sistemas adesivos, clareamento, esmalte dental, adesão.

### INTRODUCTION

A new era in dentistry began with the enamel etching technique<sup>1</sup> and the development of composite resins. Clinicians no longer need retentive preparations. Today, adhesive systems show bonding ability to enamel and dentin, and restorative methods are less invasive. In addition to these developments, there are other procedures that provide better smile esthetics. Doubtless, the

alteration of dental color has stimulated the development and improvement of stain removal techniques and whitening techniques for dentin color alterations<sup>2-5</sup>. The success of dental whitening techniques performed by professionals has been attributed to their safety, inexpensiveness and clinical results<sup>6,7</sup>. Nevertheless, in 1988, Titley et al.<sup>8</sup>, who were researching the bonding of adhesive systems to den-

tal enamel bleached with peroxide agents, observed reduced values in bonding strength in restorations done immediately after the bleaching treatment. This fact was also verified by Torneck et al.<sup>9</sup> in 1990, and Vyver et al.<sup>10</sup>, who attributed this damage to the residual oxygen present in the dental structure and also the possible histomorphological alterations which occurred in the substrate<sup>11-13</sup>.

Even with the knowledge of these negative effects, due to their whitening success, dentifrices with bleaching agents and abrasive particles have been created to remove intrinsic and extrinsic stains<sup>14,15</sup>. Dentifrices contain bleaching agents such as carbamide peroxide or hydrogen peroxide in lower concentration in conjunction with abrasives such as calcium carbonate, calcium phosphate, hydrated silica, alumina and sodium bicarbonate.

It is worth noting that despite some reports stating that dentifrices are responsible for the expected bleaching<sup>6,14,16</sup>, these products are used without any orientation or supervision by the clinician. This is a matter of concern, especially due to their abrasiveness<sup>17,18</sup>, possible enamel morphology alterations and resin bond strength reduction. These alterations are routinely observed in dental substrates exposed to highly concentrated whitening products. Dentifrices with peroxide may be used daily, and in most cases, patients are submitted to restoration procedures without respecting the minimum break needed to eliminate the negative effects of the bleaching agent. This draws attention to the knowledge of the effect of whitening toothpaste on the bond strength of the enamel-resin composite interface.

The aim of this study was to evaluate by micro-shear bond strength test, the micro-shear bond strength resulting from the use of two different adhesive systems (one containing ethanol and the other containing acetone, both solvents) applied on bovine enamel previously exposed to dentifrices with or without peroxide. Thus, the null hypothesis is that the exposition to toothpaste containing a lower concentration of peroxide does not affect the bonding of composite resins to the dental enamel in restorations.

## MATERIALS AND METHODS

Forty recently extracted intact inferior incisive bovine teeth were used. Teeth were cleaned using periodontal devices (Dental Duflex Ltda., Rio de Janeiro, RJ, Brazil) and polished with pumice,

water and Robinson brush (KG Sorensen Ind. E Com. Ltda) at low-speed. The teeth with structural defects or caries lesions on the labial surface were rejected. The specimens were stored in 0.1% neutral thymol solution.

Roots were removed using a diamond saw, (ISOMET 2000, Buehler, Lake Bluff, Illinois, USA) 2 mm below the cementoenamel junction, and their pulp residues were removed. The crowns segments were embedded in polystyrene resin, leaving the buccal surface exposed. The convexity of the enamel surfaces was reduced by polishing with 600, 1000 and 1200-aluminum oxide grit abrasive paper disks, (Carbimet Paper Disks – Buehler, Lake Bluff, Illinois, USA) under running water, producing flat enamel surfaces.

After that, the teeth were randomly divided into eight groups ( $n=5$ ), according to the dentifrice and adhesive system to be used in each group (Table 1). A 1:2 dentifrice: distilled water suspension was used (18 mL of water and 9 g of dentifrice). The samples were submersed in dentifrice suspension for fifteen minutes per day, simulating the daily time for tooth brushing. However, no mechanical brushing was used during these procedures. Immediately after each immersion in dentifrice suspension, the teeth were immersed in artificial saliva until the next dentifrice treatment, for 21 days.

Then the specimens were washed in running tap water and stored in artificial saliva (1.5 mMol/L Ca, 50 mMol/L KCl, 0.9 mMol/L PO<sub>4</sub>, 20 mMol/L buffer TRIS - TRI-Hydroxymethyl-aminomethane) at 37°C. The specimens not submitted to the dentifrice treatment (groups I and V) were stored for the entire time in artificial saliva in the oven at 37°C ± 1°C and controlled humidity.

The bonding procedures occurred 30 minutes after the last dentifrice applications. The adhesive systems and restorative material were applied according to the manufacturers' instructions.

In Groups I, II, III and IV, the enamel was etched with 37% phosphoric acid gel for 15 seconds (Scotchbond Etchant, 3M Espe, St.Paul, Minnesota, USA), rinsed with distilled water and thoroughly dried. Two layers of adhesive system Adper Single Bond 2 (3M) were applied. Air spray was used to evaporate the solvent before polymerization for 10 seconds using quartz-tungsten-halogen light (Ultraluz- Dabi Atlante, Ribeirão Preto, SP, Brazil) with an intensity output of 450 mW/cm<sup>2</sup>.

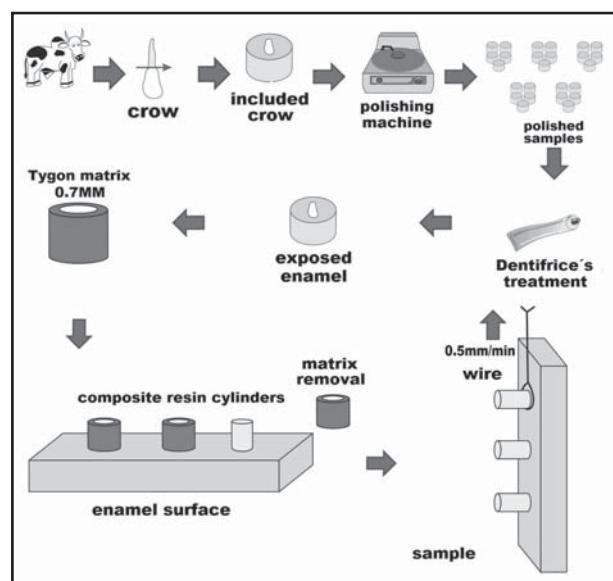
**Table 1: Experimental groups, adhesive systems and dentifrices used.**

| Group | Dentifrice              | Dentifrice trade name           | Manufacturer/<br>Dentifrice      | Adhesive system             |
|-------|-------------------------|---------------------------------|----------------------------------|-----------------------------|
| I     | -                       | -                               | -                                | Adper Single Bond 2-3M/ESPE |
| II    | Conventional            | Colgate                         | Colgate- Palmolive Co.           | Adper Single Bond 2-3M/ESPE |
| III   | With hydrogen peroxide  | Colgate Simply White Toothpaste | Colgate- Palmolive Co.           | Adper Single Bond 2-3M/ESPE |
| IV    | With carbamide peroxide | Rembrandt Plus™                 | Gilette Company, Boston, MA, USA | Adper Single Bond 2-3M/ESPE |
| V     | -                       | -                               | -                                | Prime Bond 2.1-Dentsply     |
| VI    | Conventional            | Colgate                         | Colgate-Palmolive Co.            | Prime Bond 2.1-Dentsply     |
| VII   | With hydrogen peroxide  | Colgate Simply White Toothpaste | Colgate-Palmolive Co.            | Prime Bond 2.1-Dentsply     |
| VIII  | With carbamide peroxide | Rembrandt Plus™                 | Gilette Company, Boston, MA, USA | Prime Bond 2.1-Dentsply     |

For specimens in Groups V, VI, VII and VIII, the enamel was etched with 37% phosphoric acid gel for 15 seconds (Scotchbond Etchant, 3M ESPE, St.Paul, Minnesota, USA), rinsed with distilled water and thoroughly dried. A generous amount of Prime Bond 2.1 adhesive system was applied for 15 seconds. (Dentsply Indústria e Comércio Ltda, Petrópolis, RJ, Brazil) Air spray was used to evaporate the solvent before polymerization as described for the groups above.

After adhesive curing, four hollow cylinders (1.0 mm height/0.75 mm internal diameter) were placed on the treated surfaces. A composite resin (Filtek Z350 Flow - 3M/ESPE St. Paul, Minnesota, USA) was inserted into the tube and cured for 40 seconds. Specimens with restored enamel surfaces were stored in artificial saliva and after 24 hours the tubes were removed, exposing the composite resin cylinders bonded to the enamel. Prior to the micro-shear bond strength test, the specimens were examined with a Stemi SV 11 stereoscopic microscope at 40x magnification (Carl Zeiss Company – DSM-940 A, Oberkochen, Baden – Württemberg, Germany) to ascertain whether there were any premature failures in the bonded interface, such as bubbles or material fault. Specimens were fixed in the micro-shear device and tested in a universal machine (EMIC- Equipamentos e Sistemas de Ensaio Ltda., São José dos Pinhais, PR, Brazil). The shear bond strength was applied through

an 0.20 mm (diameter) orthodontic wire fixed to the cylinder bases, at a speed of 0.5 mm/min (Fig. 1). Data were converted to MPa and the means were analyzed using two-way Anova and the Fisher's PLSD test ( $\alpha=0.05$ ). Failure modes were classified and discussed based on occurrence percentage. After the destructive test, the fragments were examined with a stereomicroscope at 66 x magnification and classified as adhesive, mixed or cohesive failure.

*Fig. 1: Schematic of methodology sequence used to produce of the micro-shear samples and test execution.*

## RESULTS

The two way ANOVA analysis showed that there was no significant interaction between the variables material and treatment; analyzing the mean value of two adhesives produced by different treatments using Fisher's PLSD test.

Considering Table 2, no statistically significant difference was found between the mean values of each adhesives system ( $p=0.3527$ ). In the dentifrice treatments there were statistically significant differences between micro-shear bond strength means of the control and carbamide groups ( $p=0.0145$ ) and the control and hydrogen groups ( $p=0.0370$ ). The mean micro-shear bond strength of samples submitted to conventional toothpaste showed no statistical difference compared to any other group.

Fig. 2 shows the failure mode classifications. Adhesive fractures were frequently identified in all groups, except group IV, which showed predominance of cohesive fracture modes in enamel. Specimens treated with Prime & Bond 2.1 showed a higher percentage of cohesive fracture modes in enamel compared to the groups treated with Adper Single Bond, except group IV.

## DISCUSSION

Dental whitening techniques are achieving unprecedented success in dentistry. However, the lack of basic knowledge for safe treatment may be harmful to tooth structure.

On the one hand, aggressive marketing and non-specialized publications contribute to the information process and the general population learns of esthetic dentistry's current resources. On the other hand, it creates a disorderly search for this kind of treatment, which is technically simple, but not yet clearly explored biologically.

As whitening procedures are so successful, peroxide agents are being added to antiseptic solutions, mouth rinses and toothpastes. Although the peroxide concentration in these products is lower than the concentration used in trays (home-use) or office bleaching techniques, data confirming their oxidant action have already been published<sup>3,8,10,19</sup>.

Whitening products are responsible for great damage in restoration performance, when done immediately after bleaching treatment<sup>22</sup>. In this context, increase in microleakage, decrease in microhardness, decrease in tag length and even pigmentation of the restorations are being reported<sup>19,20-23</sup>.

Having free access to whitening dentifrices, people are often not suitably warned by clinicians about the possible harmful action of these products on restorative procedures.

In this study, although there is similar adhesive performance, it was also found that the treatment of a substrate with peroxide dentifrices caused a significant reduction in bonding values when compared to the control group (baseline).

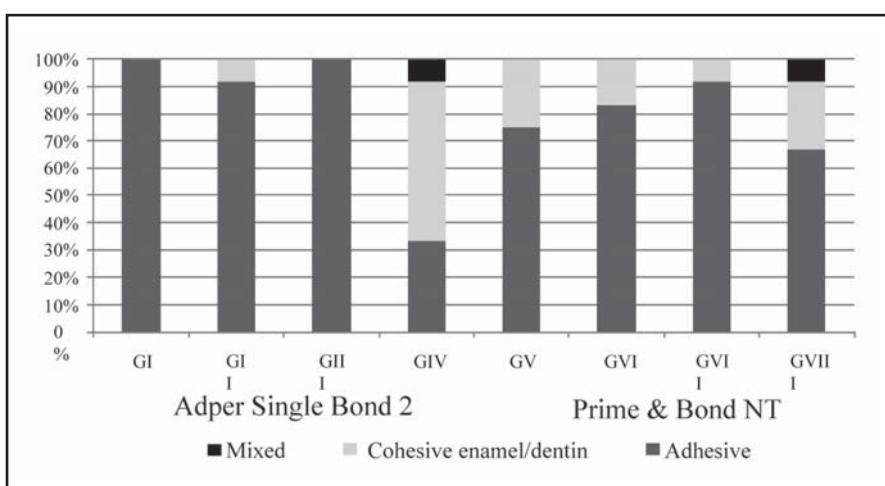


Fig. 2: Failure modes after micro-shear bond strength test.

**Table 3.1: Micro-shear bond strength values.**

|              | Control            | Conventional        | Carbamide          | Hydrogen           | mean               |
|--------------|--------------------|---------------------|--------------------|--------------------|--------------------|
| Prime & Bond | $26.03 \pm 1.94$   | $25.38 \pm 3.20$    | $25.16 \pm 4.71$   | $24.22 \pm 2.70$   | $25.20 \pm 3.14$ a |
| Single Bond  | $26.71 \pm 5.14$   | $25.49 \pm 5.27$    | $21.89 \pm 3.25$   | $23.70 \pm 4.08$   | $24.45 \pm 4.44$ a |
| Mean         | $26.37 \pm 3.54$ a | $25.44 \pm 4.24$ ab | $23.53 \pm 3.98$ b | $23.96 \pm 3.39$ b |                    |

Comparing the treatments, a statistically significant difference was found between control/carbamide groups, and control/hydrogen groups, suggesting possible negative interactions in the bond strength to enamel after receiving bleaching products, independently of the adhesive system used.

Previous studies confirmed possible interference of the bleaching agents with mechanical and morphological features in the bleached teeth's bonding interface, with alterations of bond strength values and failure modes between the adhesive and the bleached enamel<sup>19,20,24</sup>.

The study results do not agree with the data in literature, which show that after carbamide peroxide dentifrice use, there is an increase in shear bond strength<sup>3</sup>. This difference between the results may be related to the different methodology applied. This study used a micro-shear bond strength test, which analyses minor regions, and is therefore more sensitive to slight alterations and also more representative<sup>25</sup>. Furthermore, this study used a solution with a higher proportion of dentifrice (2:1), which may have propitiated an increase in the oxygen concentration at the enamel surface and major surface changes in the organic and inorganic matrix<sup>26,29</sup>.

Although cohesive failures in enamel occurred, probably caused by hydrogen peroxide action, the failure analyses showed a higher number of adhesives failures, demonstrating that micro-shear bond strength test was performed correctly (Fig. 2).

The oxygen remaining in the dental tissues after bleaching agent applications can impede the appropriate polymerization of the adhesive and

thus contribute to the reduction of bond strength values<sup>3,8,10</sup>.

Furthermore, it has been suggested that alterations occurring in the protein and mineral components of the surface of the enamel layer might be responsible for the reduction in bonding strength<sup>26,27</sup>. A possible manifestation of this effect is noted in the failure mode analysis, as the samples submitted to bleaching dentifrices showed major cohesive failure in the enamel, suggesting major fragility of the substrate (Fig. 2). In addition, the samples submitted to the hydrogen peroxide product showed an increase in the quantity of fractures in the enamel compared to the carbamide peroxide dentifrice, which might be explained by the fact that the carbamide products are degraded to release hydrogen peroxide, producing a bleaching active principle of lower concentration<sup>4,28</sup>. In this study, the adhesive performance was similar in different treatments. This may be related to the utilization of the all-etch technique in enamel, providing excellent performance, because of its demineralization, complete removal of the smear layer and lower humidity after etching<sup>29</sup>. Literature describes that the use of acetone adhesives may provide better results in recently bleached teeth<sup>20</sup>. However, the utilization of adhesives with different solvents did not influence the result, demonstrating that this result was influenced by the dentifrice used, rejecting the null hypothesis.

## CONCLUSION

The adhesive procedure may be harmed when performed immediately after the use of dentifrices containing hydrogen peroxide or carbamide peroxide.

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## CORRESPONDENCE

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