

# The Influence of Time Interval between Bleaching and Enamel Bonding

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## ABSTRACT

**Objectives:** The purpose of this study was to investigate the penetration of a conventional adhesive material into enamel bleached with 16% carbamide peroxide and 38% hydrogen peroxide using optical light microscopy.

**Methods:** Extracted human teeth were randomly divided into eight experimental groups with six specimens each, according to the bleaching material and time interval after bleaching and before the bonding procedure. Groups were designated as follows: control group, restorations in unbleached teeth; restorations performed immediately after bleaching; restorations performed 7 days after bleaching; restorations performed 14 days after bleaching; and restorations performed 30 days after bleaching. The length of resin tags was measured with an Axiophot photomicroscope at 400× magnification for the calculation of the proportion of tags of study groups compared to the respective control groups. Analysis of variance was applied for comparison between groups; data were transformed into arcsine ( $p < 0.05$ ).

**Results:** The specimens of experimental groups, in which restorations were performed 7, 14, and 30 days after bleaching, showed better penetration of adhesive material into enamel than specimens restored immediately after bleaching. There was no statistically significant difference between the bleaching materials employed or in the interaction between bleaching agent and time interval.

**Conclusions:** This suggests that a time interval of at least 7 days should be allowed between enamel bleaching and placement of adhesive bonding agents for accomplishment of composite resin restorations.

## CLINICAL SIGNIFICANCE

Establishment of adequate time after bleaching is fundamental to allow the normal penetration of a one-bottle conventional adhesive onto the enamel surface.

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## INTRODUCTION

Haywood and Heymann,<sup>1</sup> searching for more conservative procedures in 1989, reported on a bleaching technique called “home-bleaching,” which employed a 10% carbamide peroxide gel for bleaching of discolored teeth. Currently, concentrations ranging from 10 to 20% have been used for its accomplishment,<sup>2</sup> as well as other bleaching techniques called in-office techniques, which employ highly concentrated products with hydrogen peroxide at concentrations of 30 to 38%.<sup>3</sup>

The bleaching treatment encourages patients to accept additional esthetic treatments. When patients have their teeth bleached, they often become interested in replacement of old restorations, closure of diastemas, or other esthetic procedures.<sup>4</sup> In addition, bleaching may be indicated before placement of an esthetic resin restoration to obtain a more pleasing final shade for the case.<sup>5</sup> However, some studies have shown that the bond strength of bonded restorations to tooth structures is reduced when the tooth has been bleached, using either an in-office or at-home technique.<sup>5–10</sup> Clinically, this reduction in bond strength is pertinent because vital bleaching is often considered as the first step to improve the appearance of teeth prior to applying a bonded restoration.<sup>5</sup>

Some authors speculate that residual peroxide and/or oxygen radicals

in bleached teeth may interfere with the polymerization of adhesive restorative materials and therefore decrease the bond strength.<sup>6–9,11</sup> On the other hand, other authors report that changes in proteins and mineral content of the most superficial enamel layers may also be responsible for the reduced bond strength.<sup>10,12–14</sup>

There seems to be a consensus on the effects of tooth bleaching performed before bonding procedures, both with the utilization of hydrogen peroxide and carbamide peroxide.<sup>9</sup> However, it is not known if the concentration and/or type of bleaching agent and time interval between bleaching and the bonding procedure may influence the bond achieved.

Studies showed that resin tags in bleached enamel subsequently etched with 37% phosphoric acid were less defined, more fragmented, and penetrated into a lesser depth.<sup>11,15</sup> Decreased bond strength has also been reported, because the quality of the composite bond is affected by the decreased number of resin tags.<sup>16</sup>

There is little evidence from reports in the literature concerning the interface between the enamel and resin and resin tag penetration in bleached enamel when different bleaching agents are compared.

The purpose of this study was to analyze the penetration of an adhesive material when applied on enamel submitted to bleaching with 16% carbamide peroxide (Clarigel Gold 16%, Dentsply Indústria e Comércio Ltda, Petrópolis, RJ, Brazil) and 38% hydrogen peroxide (Opalescence Xtra Boost, Ultradent Products, Inc., Salt Lake City, UT, USA), by light microscopy analysis. The penetration of bonding agents was evaluated after various time intervals between bleaching and restorative treatment. The null hypothesis was that there is no difference in the penetration of a conventional one-step adhesive when applied on enamel submitted to bleaching with 16% carbamide peroxide and 38% hydrogen peroxide at different time intervals between bleaching and restorative treatment.

## MATERIALS AND METHODS

The study was approved by the Institutional Review Board of Araraquara Dental School, University of the State of São Paulo (Araraquara, SP, Brazil).

The study sample was composed of 48 intact human premolars obtained from patients aged 14 to 20 years. Specimens were randomly divided into eight experimental groups ( $N = 6$ ) according to the bleaching materials and time intervals after bleaching before accomplishment of the bonding

procedure. All specimens in each group had their roots embedded in a single acrylic resin block, keeping only the coronal portion exposed.

The control group was designed on three buccal aspects and three lingual aspects of specimens in each group, whereas the other aspects were submitted to the experimental conditions proposed. These aspects were identified by the creation of a groove in the enamel at the level of the cemento-enamel junction using a high-speed carbide bur (1/2, K.G. Sorensen Ind & Com, Alphaville, São Paulo, SP, Brazil). Thus, each specimen presented a control aspect and an experimental aspect (Fig. 1).

Before tooth bleaching, the control aspects were submitted to prophylaxis with pumice and water, etching with 37% phosphoric acid for 1 minute,<sup>15,17</sup> washing for 15 seconds, and drying. Three coats of the adhesive system Prime Bond 2.1 (Dentsply Indústria e Comércio Ltda.) were applied, gently air-

dried, and light-cured for 20 seconds (Sybron Kerr, Danbury, CT, USA). A 2-mm-thick layer of composite resin ESTHET-X (Dentsply Indústria e Comércio Ltda.) was applied and light-cured for 40 seconds.

Specimens were immersed in artificial saliva and stored in an oven at 37°C for 1 week before application of the bleaching agents Clarigel Gold 16% (GI) or Opalescence Xtra Boost (GII) on the buccal or lingual experimental aspects of each corresponding study group. Time intervals varied between groups, as follows: T1 (immediate), T2 (7 days), T3 (14 days), and T4 (30 days). The composition of the aforementioned products is presented in Table 1.

Specimens in groups GIT1, GIT2, GIT3, and GIT4 were submitted to bleaching with carbamide peroxide Clarigel Gold 16%. Before bleaching, impressions were taken with alginate for fabrication of dental

casts, on which custom trays were fabricated with thermoplastic material. At the region corresponding to each tooth, one drop of bleaching material was inserted in each tray, combined with a drop of artificial saliva (Table 2); these were placed in position and stored for 4 hours in an oven at 37°C. Then, the trays were removed and the teeth were thoroughly washed with air-water sprays and immersed once again in artificial saliva for 20 hours until the following application of bleaching agents. Fourteen applications of the bleaching agent Clarigel Gold 16% were performed.

During the period of bleaching treatment, the teeth were brushed with a soft-bristle toothbrush twice a day, before and after application of the product, with a low-abrasive toothpaste (Opalescence Cool Mint, Ultradent Products, Inc.).

Application of the bleaching agent Opalescence Xtra Boost in specimens of groups GIIT1, GIIT2, GIIT3, and GIIT4 was performed after mixture of the bleaching material with its activator. The material was applied on the experimental aspects of specimens and slightly extended to the cusp inclines. The material was left on the tooth surface for 10 minutes; at 5 minutes of application, the material was shaken with a dental probe. Then, the teeth were washed with air-water spray and air-dried. For this

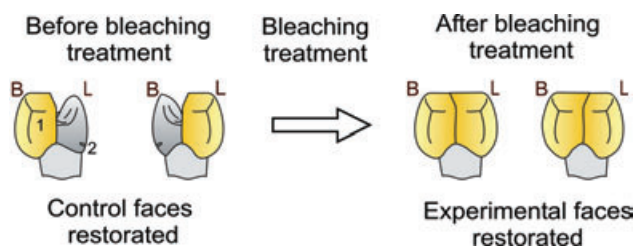


Figure 1. Scheme of sequence of the restoration of the control and experimental aspects. (1) Control aspect restored with composite resin. (2) Mark for identification of experimental aspect after lingual (L) and buccal (B) sectioning.

**TABLE 1. MATERIALS EMPLOYED ACCORDING TO COMMERCIAL BRAND AND MANUFACTURER.**

Material	Commercial Brand	Manufacturer	Composition
Tooth bleaching system (GI) 16% carbamide peroxide	Clarigel Gold 16%	Dentsply Batch: 4155	Sodium benzoate, propylene glycol, carbopol, carbamide peroxide, sodium fluoride, distilled water, and mint fragrance.
Tooth bleaching system (GII) 38% hydrogen peroxide	Opalescence Xtra Boost	Ultradent Batch: 015	38% hydrogen peroxide, EDTA, sodium fluoride, photoinitiators.
Etching agent	37% phosphoric acid	FGM	—
Adhesive	Prime & Bond 2.1	Dentsply Batch: 5029	Dipentaerythritol pentaacrylate phosphate, cetylamine hydrofluoride, acetone, photoinitiators and stabilizers.
Composite resin	ESTHET X	Dentsply Batch: 0110	Urethane-modified Bis-GMA, Bisphenol-A, ethoxylate dimethacrylate (Bis-EMA), and TEGDMA, silanized fluoride, and barium aluminium borosilicate glass fillers with mean size smaller than 1 µm and colloidal silica measuring 0.04 µm and nanometric silica.

Bis-GMA = bisphenol A-diglycidylmethacrylate, EDTA = ethylene diamine tetraacetic acid, TEGDMA = triethyleneglycol dimethacrylate.

**TABLE 2. COMPOSITION OF ARTIFICIAL SALIVA, MODIFIED FROM BO KRASSE,<sup>20</sup> USED IN THE PRESENT STUDY (pH 7.0).**

Components	Concentration (in 1 L of solution)
Carboxymethylcellulose	4 g
Sorbitol	60 g
Potassium chloride	1 g
Sodium chloride	1 g
Sodium fluoride	2 mg
Magnesium chloride	50 mg
Calcium chloride	150 mg
Potassium phosphate	400 mg
Nipagin	2 mg
Distilled water	Add sufficient water to produce 1,000 mL

bleaching agent, three applications were performed in a single session, without application of a light source.

Immediately after the last application of each bleaching material, the specimens in experimental groups

GIT1 and GIIT1 were submitted to the bonding procedures on the experimental aspects as performed for the control aspects.

At the other time intervals T2, T3, T4, in both experimental groups (GI and GII), the bonding proce-

dures were applied at 7, 14, and 30 days after bleaching, respectively, following the same methodology described for the control group.

Specimens were sectioned into five buccolingual plane sections with a thickness of nearly 200 µm (Isomet 2000, Buehler UK Ltd., Lake Bluff, IL, USA). Three sections were selected and further polished with aluminum oxide abrasive paper with grit #360 and #600 to a thickness of approximately 100 µm (Figure 2). Sections were decalcified in 40% nitric acid for approximately 60 seconds, ie, until the enamel was dissolved, maintaining the adhesive with resin tags. Specimens were then immersed in distilled water, mounted on glass, and covered by a glass cover slide. Edges were sealed with Canadian oil.

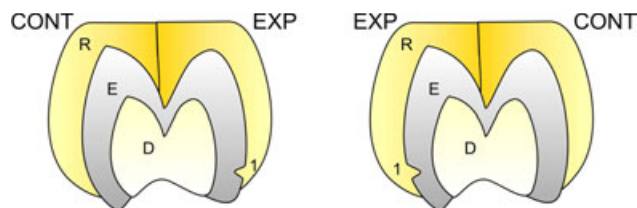


Figure 2. Scheme of longitudinal section by wearing of specimens (100 µm), exhibiting the control (CONT) and experimental (EXP) aspects.  
R = resin; E = enamel; D = dentin; 1 = mark for identification of experimental aspect.

TABLE 3. MEANS AND SD OF THE PROPORTION OF ADHESIVE PENETRATION IN EXPERIMENTAL GROUPS COMPARED WITH PENETRATION IN THE CONTROL GROUP.

Bleaching Agent	Groups	Mean	SD
GI Clarigel Gold 16% (carbamide peroxide)	T1 (immediate)	0.52	0.13
	T2 (7 days)	0.74	0.22
	T3 (14 days)	0.75	0.21
	T4 (30 days)	0.85	0.05
GII Opalescence Xtra Boost (hydrogen peroxide 38%)	T1 (immediate)	0.54	0.12
	T2 (7 days)	0.69	0.09
	T3 (14 days)	0.91	0.06
	T4 (30 days)	0.87	0.11

TABLE 4. ANALYSIS OF VARIANCE OF THE PROPORTION OF ADHESIVE PENETRATION IN EXPERIMENTAL GROUPS COMPARED WITH PENETRATION IN THE CONTROL GROUP.

Source of Variation	DF	Sum of Squares	Mean Square	F	Pr > F
Bleaching agent	1	0.00006400	0.00006400	1.25	0.2703
Time	3	0.00292122	0.00097374	19.01	<0.0001*
Bleaching agent/time	3	0.00019632	0.00006544	1.28	0.2951
Residue	40	0.00204864	0.00005122		
Total	47	0.00523018			

DF = degrees of freedom, F = value test, Pr = probability test

\*Statistically significant.

Data were transformed into arcsine.

The sections of each tooth were analyzed and measured on a Axio-phot light microscope (Carl Zeiss DSM-940 A, Zeiss, Oberkochen,

Germany) at 400× magnification, with a micrometric ocular of 40/075. Measurements of the resin tags of each section at the buccal

and lingual sides were performed by careful analysis of the entire extension of each section, blindly conducted by a single calibrated examiner. Three measurements of resin tags were performed and collected at three different points on each surface, namely, at the occlusal, middle, and cervical thirds of each section. The mean of each specimen corresponded to the proportion of resin tags of the experimental aspect compared with the resin tags of the control aspect at completion of each analysis.

The proportional means were submitted to two-way analysis of variance (ANOVA) with a transformation of proportion in arcsine to confirm the normal distribution and homocedasticity of groups. When the significance was found, Tukey test was applied at a significance level of 5%.

## RESULTS

A two-way ANOVA, applied to the means transformed into proportions of length of resin tags at the control and experimental aspects (Table 3), revealed significant difference according to time interval before the restorative procedures. There was no statistically significant difference between the bleaching materials employed and the interaction between bleaching agent and time interval (Table 4).

Time intervals of 7, 14, and 30 days after tooth bleaching allowed

higher mean penetration of the adhesive system into the enamel surface than when the adhesive system was applied immediately after bleaching (Figure 3). The mean penetration at 7 days was higher compared with the immediate accomplishment of the restorative procedure and statistically similar to the 14-day period. The mean penetration was also similar at 14- and 30-day intervals (Figure 4 and Table 5).

#### DISCUSSION

The literature has reported that bonding to recently bleached enamel may be impaired, regardless of the type and concentration of bleaching agent.<sup>5,6,8-10,15</sup> The present study revealed reduced penetration of the adhesive system, with

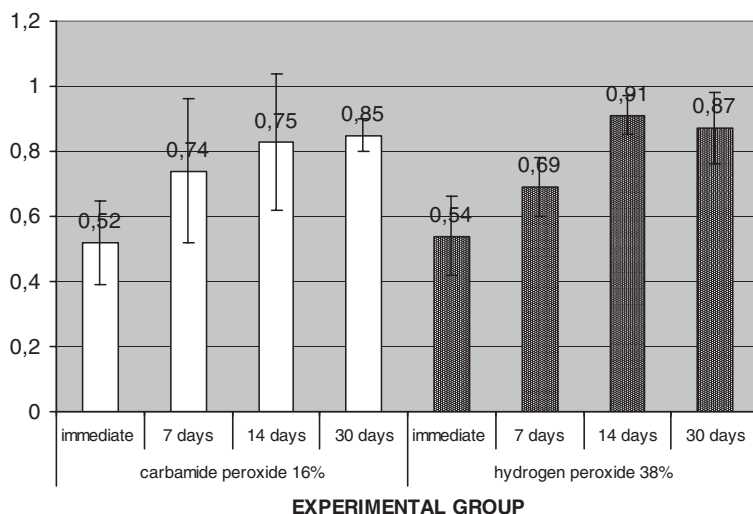
formation of small and irregular tags in the groups restored immediately after completion of the bleaching treatment. Several hypotheses have been presented to explain this phenomenon; one of them mentions that bonding may be impaired by mineral, protein, and structural changes in the enamel.<sup>10,12-14</sup> However, this study

revealed that penetration of the adhesive system into dental enamel was considerably increased at 7 days after bleaching. Thus, it may be suggested that the utilization of artificial saliva, which has a remineralizing potential, may have allowed the reorganization of the morphological changes occurring in the enamel,<sup>15,18,19</sup> thus allowing an

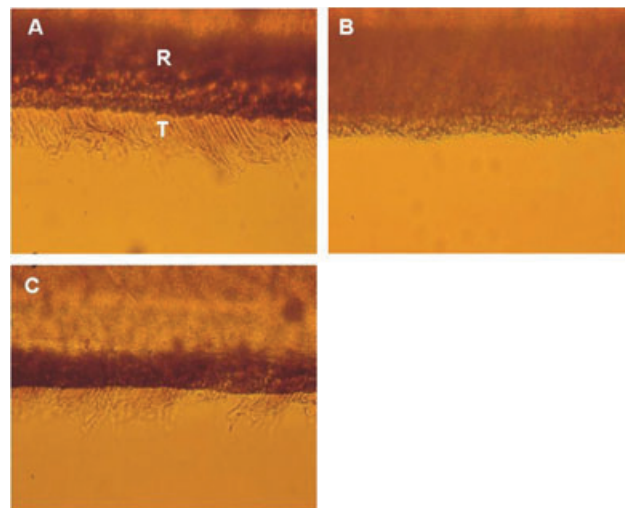
**TABLE 5. TUKEY TEST OF THE PROPORTION MEANS OF ADHESIVE PENETRATION IN EXPERIMENTAL GROUPS COMPARED WITH PENETRATION IN THE CONTROL GROUP FOR EACH STUDY PERIOD.**

Mean	N	Time	Result of the Test
0.85	12	T4 (30 days)	a
0.82	12	T3 (14 days)	ab
0.71	12	T2 (7 days)	b
0.53	12	T1 (immediate)	c

Means with different letters are statistically different. Significance level 5%.



**Figure 3.** Proportional means of resin tags for bleaching materials according to the time interval before application of an adhesive material on the enamel surface.



**Figure 4.** Longitudinal sections by wearing (100  $\mu$ m) of the enamel-resin interface. A, Photomicrograph of the interface of one specimen from the control group. B, Interface achieved with application immediately after bleaching. C, Interface achieved at 14 days after bleaching. R = resin; T = resin tags.



etching pattern compatible with a good penetration of the adhesive system. According to Josey and colleagues in 1996,<sup>14</sup> etching soon after bleaching with hydrogen peroxide led to the loss of prismatic form, and the enamel seemed to be overetched.

On the other hand, some authors have reported the influence of by-products on the loss of adhesive properties.<sup>6-9,11</sup> Bleaching products containing carbamide peroxide are decomposed into some by-products as ammonia, urea, and carbon dioxide after contact with humidity, besides free radicals that originated from the breakage of hydrogen peroxide from carbamide peroxide.<sup>1</sup> Products containing high concentrations of hydrogen peroxide produce only free radicals that originated from the breakage of peroxide, and thus the possibility of interference of by-products of carbamide peroxide (ammonia, urea, and carbon dioxide) on the formation and penetration of resin tags may be discarded because groups GI and GII were not statistically different.

The main by-products of hydrogen peroxide are water and oxygen.<sup>1</sup> According to some authors, the oxygen might interfere with the polymerization of adhesive systems,<sup>5-11</sup> which are cured by a free radical mechanism.<sup>18</sup> Scanning electron microscope examinations of the enamel revealed the presence

of a granular, more porous resin and evidence of bubbling, which could be the result of an oxidizing reaction because of the entrapment of peroxide along interprismatic spaces.<sup>8,11</sup> Once the entrapped peroxide is eliminated (during the time period allowed before the accomplishment of bonding procedures), the enamel surface shows increased bonding as a result of the reduction in surface and subsurface contaminants, which in turn results in more effective etching and resin penetration.<sup>11</sup> These facts were demonstrated in groups restored after 7 days, which is enough time for eliminating most of the oxygen. These findings have been previously reported; the elimination of these residual products over time probably occurs in a period of 1 to 3 weeks after completion of the bleaching treatment.<sup>9,14,15</sup>

Perdigão and colleagues<sup>10</sup> did not find differences in the amount of oxygen present on bleached and nonbleached enamel surfaces. However, the hypothesis of the interference of oxygen on bonding is increasingly explained by studies using antioxidants, which allow the achievement of similar bond values immediately after bleaching compared with the control group.<sup>8,19</sup> However, clinical utilization of antioxidants is still unknown. The present study revealed that the increased time interval before the accomplishment of bonding procedures, ie, at 7, 14, and 30 days after

bleaching treatment, allowed better penetration of the adhesive, possibly because of the elimination of residual peroxides.

As a result of the influence of tags on enamel bonding,<sup>16</sup> an association can be made with studies observing the reduction of bond strength in teeth immediately bleached with different products. The increase in the time period allowed before the accomplishment of bonding procedures increases the bond strength up to similar values as that of the control group.

In "in vivo" conditions, the oral kinetics may reduce this interval required for the elimination of oxygen and reorganization of the enamel. However, a time interval compatible with laboratory studies would be favorable because few clinical studies have been conducted to evaluate the durability of restorations performed after bleaching by both techniques.

## CONCLUSION

The accomplishment of restorative procedures immediately after completion of bleaching significantly interfered with the penetration of adhesive agents into the enamel surface, suggesting that a period of at least 7 days should be allowed between the utilization of peroxide bleaching material and restorative procedures that require acid-etching and adhesive bonding materials.

DISCLOSURE AND  
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