Shear Bond Strength of Enamel Treated with Seven Carbamide Peroxide Bleaching Agents

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ABSTRACT

Purpose: Lower average values of bond strength of adhesive systems to enamel bleached with 10% carbamide peroxide agents have been reported, but the effects of higher concentrations of carbamide peroxide bleaching agents are still unknown. The aim of this in vitro study was to evaluate the shear bond strength of enamel treated with different concentrations of carbamide peroxide to an adhesive system after a postbleaching period of storage in artificial saliva for 15 days.

Materials and Methods: Seven carbamide peroxide bleaching agents with concentrations varying from 10 to 22% were analyzed. A placebo agent was used as a control group. The agents were applied on the enamel fragments for 8 h/d for 42 days. During the remaining time, the specimens were stored in artificial saliva. After that time, the fragments were stored individually in artificial saliva for 15 days. An adhesive system was used to bond resin-based composite cylinders on the enamel surface. Shear bond strength tests were performed and the fractured surfaces of the specimens were visually examined with a stereomicroscope at $\times 30$.

Results: The analysis of variance did not show differences in shear bond strength among the treatment agents. The fractures for all treatment agents were predominantly adhesive.

Conclusion: After 15 days storage in artificial saliva, different concentrations of carbamide peroxide bleaching agents and a placebo agent had the same enamel shear bond strength values.

CLINICAL SIGNIFICANCE

Concentrations varying from 10 to 22% carbamide peroxide agents cause no differences in shear bond strength of enamel to an adhesive system after 15 days of storage in artificial saliva.

(*J Esthet Restor Dent* 16:250–260, 2004)

C ince its introduction by Haywood and Heymann in 1989,1 nightguard vital bleaching has been a procedure that has grown dramatically in popularity in the dental offices because of its efficiency and simplicity to remove intrinsic stains from the teeth.² Modifications, improvements, and

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variations of the technique have been introduced, including the use of different concentrations of carbamide peroxide agents (10–22%) with carbopol and the use of these agents for one or more intervals during the day.^{2,3} Some manufacturers and clinicians have claimed that the use of higher concentrations of carbamide peroxide bleaching agents is more effective, and that whitening results can be achieved more quickly than with the use of a 10% carbamide peroxide agent.²

The decomposition products of carbamide peroxide-hydrogen peroxide and urea-are obtained after the dissociation of the bleaching agent in contact with saliva and oral fluids. Urea is degraded into ammonia and carbon dioxide. The hydrogen peroxide, owing to its instability, decomposes into water and oxygen. The oxygen penetrates through the pores of the enamel and dentin to provide lightening of the teeth. Through oxidation reactions, the large pigmented molecules in enamel and dentin are opened and converted into smaller pigmented molecules, which are lighter in color.4 In some cases, restorations will need to be changed because their color is not clinically acceptable with the color attained by the natural teeth. When esthetic restorations are needed, they should be placed using a bonding technique^{5,6}; however, it is unknown whether immediate bonding of resin after bleaching procedures decreases the bond strength of some of these restorative materials.7

Some studies have shown significantly lower average values of bond strength of composite resin to bleached enamel compared with unbleached enamel.^{8–18} Others have reported no differences in bond strength values after exposing the enamel specimens to artificial saliva during or after the bleaching treatment.^{19,20}

The residual peroxide and the oxygen have both been claimed as factors that affect the polymerization of the adhesive systems and composite resins.7-21 Resin tags in bleached enamel are less numerous, shorter, and less defined than those in unbleached enamel,9,10,12,15 and there has also been evidence of bubbling.11,13 These changes seem to be time and concentration dependent.13,15,22 Changes in proteins and in mineral content of the most superficial layers of enamel may also be responsible for the reduced bond strengths.7

Methods have been indicated to counteract adverse effects related to the lower bond strength values that follow bleaching. The delaying of bonding procedures until 24 hours to 2 weeks after bleaching is the most common recommendation.^{8,12,15,16,23} The removal of superficial enamel has been suggested to restore bond strengths to normal levels,²⁴ and the use of solvents, acetone-based adhesive systems, or antioxidants has been proposed to result in less compromised composite bond strength when restorative procedures are to be completed immediately after bleaching.7,18,21,25 However, the effects of higher concentrations of carbamide peroxide bleaching agents on shear bond strength of adhesive systems to enamel are still unknown. Despite the use of methods to avoid the lower bond strength values, higher concentrations of carbamide peroxide agents could release higher amounts of residual peroxide products on enamel and/or dentin, inhibiting the polymerization process. Even when the specimens are maintained for 15 days in artificial saliva previous to the procedures of bonding, lower bond strength values may occur. Changes in the mineral content and in proteins are also related in a higher proportion.^{3,7,26,27} Therefore, the aim of this in vitro study was to evaluate the shear bond strength of an adhesive system to enamel treated with different concentrations of carbamide peroxide at a postbleaching period of storage in artificial saliva for 15 days.

MATERIALS AND METHODS

Preparation of the Dental Fragments

The study protocol was reviewed and approved by the local ethics committee. Fifty nonerupted third molars freshly extracted and stored in 10% formaldehyde (pH 7.0) were used. The teeth were submitted to a soft tissue débridement with periodontal curettes and cleaned with slurry of pumice stone in a webbed rubber cup in a slow-speed handpiece. The roots were removed approximately 2 to 3 mm apical to the cementoenamel junction. The crowns were longitudinally sectioned to obtain the dental fragments using doublefaced diamond disks in a lowspeed motor. Care was taken not to leave the dental fragments dehydrated for a long period of time. Afterward, the dental fragments were soaked in distilled and deionized water at 37°C.

The fragment sizes were required to be > 4 mm long \times 4 mm wide \times 3 mm deep, and those that presented stains or cracks after the observation on stereoscopic loupe were discarded. One hundred twenty dental fragments were embedded individually in a self-curing polyester resin in a PVC ring mold of almost 2 cm diameter, with the external surface of the enamel exposed. The molds were removed and all the external surfaces of the dental fragments were leveled by a water-cooling mechanical grinder (Maxgrind[®], Solotest São Paulo, SP, Brazil) using 400-, 600-, and 1,000-grit Al₂O₃ disks (Carborundum[™], 3M of Brazil Ltd., Recife, PE, Brazil) refrigerated with water. These procedures were conducted to obtain flat standardized surfaces for the shear bond strength tests.

A uniform area of 9 mm² (3 mm long \times 3 mm wide) of exposed enamel was created on the speci-

mens by covering the remaining dental fragment with two coatings of nail varnish. Afterward, the specimens were randomly assigned to eight groups (n = 12) to receive the different treatment agents.

Specification of the Materials

In this study, various concentrations of carbamide peroxide bleaching agents were evaluated: Nite White® 10% Excel (Discus Dental, Inc., Los Angeles, CA, USA); Nite White® 16% Excel (Discus Dental Inc.); Nite White[®] 22% Excel (Discus Dental Inc.); Opalescence[™] 10% (Ultradent Products Inc., South Jordan, UT, USA); Opalescence™ PF 20% (Ultradent Products Inc.); Rembrandt® 15% (Den-Mat Corporation, Santa Maria, CA, USA); Nupro Gold® 10% (Dentsply Preventive Care, New York, PA, USA). The control group consisted of a placebo agent prepared with carbopol and glycerin (Proderma-Pharmacy, Piracicaba, Brazil). The color and consistency of the placebo agent were similar to one of the bleaching agents (Opalescence 10%), but the placebo was pH neutral and had no active ingredient (carbamide peroxide). Table 1 presents the basic composition, lot number, pH level, and the manufacturer of each treatment agent.

Exposure of the Dental Fragments to the Bleaching Materials

A small individual tray was manufactured for each specimen using a flexible ethyl vinyl acetate polymer (Bio-Art Dental Equipment Ltd., São Carlos, SP, Brazil) that was 0.4 mm thick.

The enamel fragments were subjected to the treatment agents (experimental and control) for 8 h/d for a period of 42 days. Although bleaching results are generally seen in 2 to 3 weeks, Haywood and Heymann reported that final outcome is complete in 5 to 6 weeks (42 d).¹

For the application of the treatment materials, 0.02 mL of each agent was applied to each specimen using a calibrated syringe. Afterward, the specimens were individually covered with the tray and placed into individual closed containers with 13.5 mL of artificial saliva at 37°C.

After 8 hours, the tray was removed and the treatment agents were washed from the surface of the specimens under running distillated and deionized water for 5 seconds. During the remaining time (16 h/d), the specimens were maintained in individual closed containers with 13.5 mL of artificial saliva at 37°C. The artificial saliva in the containers was changed daily, placing new solution in the containers after removing the treatment agents from the specimens. The use of artificial saliva was proposed by Featherstone and colleagues and described by Serra and Curv.^{28,29} After the treatment period of 42 days, the specimens were placed in individual

Agent (Lot No.)	Composition	рН	Manufacturer and Location
Nite White 10% Excel (NW10) (peppermint) (9FK)	10% carbamide peroxide, polyethylene glycol, propylene glycol, hydroxypolycellulose, carbopol, flavor, sodium hydroxide	7.49	Discus Dental, Inc., Los Angeles, CA, USA
Nite White 16% Excel (NW16) (peppermint) (8HJ)	16% carbamide peroxide, polyethylene glycol, propylene glycol, hydroxypolycellulose, carbopol, flavor, sodium hydroxide	7.46	Discus Dental, Inc.
Nite White 22% Excel (NW22) (peppermint) (9KD)	22% carbamide peroxide, polyethylene glycol, propylene glycol, hydroxypolycellulose, carbopol, flavor, sodium hydroxide	7.84	Discus Dental, Inc.
Opalescence 10%* (OPA10) (regular) (3MPL)	10% carbamide peroxide, carbopol, glycerin, flavoring	6.68	Ultradent Products Inc., South Jordan, UT, USA
Opalescence PF 20% (OPA20) (regular) (3MTJ)	20% carbamide peroxide, carbopol, glycerin, flavoring, 3% potassium nitrate, 0.11% ion fluoride	6.70	Ultradent Products Inc.
Rembrandt 15% (REM15) (regular) (030371545)	15% carbamide peroxide, glycerin, sodium citrate, carbopol, flavor, triethanolamine	6.22	Den-Mat Corporation, Santa Maria, CA, USA
Nupro Gold 10% (NG10) (regular) (9911021)	10% carbamide peroxide, glycerin, carbopol, flavor	6.24	Dentsply Preventive Care, New York, PA, USA
Placebo	5% glycerin, 1.2% carbopol	7.0	Mixed formula, Proderma— Pharmacy, Piracicaba, Brazil

containers with artificial saliva at 37°C for 15 days.

Shear Bond Strength Tests

For the shear bond strength tests, the specimens were removed from the artificial saliva and the bonding area of each fragment was demarcated by placing a piece of vinyl tape, in which a 2.5 mm diameter hole had been punched, over the enamel surface. The acid etching was then performed with 35% phosphoric acid (3M ESPE Dental Products, Sumaré, SP, Brazil) for 30 seconds. The etchant was rinsed for 15 seconds under running tap water, and the enamel was dried with a gentle stream of compressed air for 5 seconds. Subsequently, two consecutive coats of an ethanolbased adhesive system (Single Bond, 3M ESPE Dental Products) were applied with a saturated brush tip. After gently air drying for 5 seconds, the material was light cured for 10 seconds, according to the manufacturer's directions.

A 5 mm high and 2.5 mm diameter polytef ring mold was placed against

the specimen to receive a filling material (Filtek Z250[™], 3M ESPE Dental Products). Two separate increments were placed and light cured for 40 seconds (Optilux 500[®], Demetron, Danbury, CT, USA). The ring mold was then removed and the composite resin was light cured for another 20 seconds on each of the opposite sides of the resin cylinder. Curing light intensity was monitored after every five specimens with a curing radiometer connected to the light-curing unit. The light intensity ranged from 550 to 650 mW/cm². The restorative

Groups	n	Mean Value (MPa)	SD (MPa)	Range (MPa)	p < .05*
Nite White 10% Excel	12	15.75	4.23	6.57-21.65	а
Nite White 16% Excel	12	16.19	6.14	7.5-27.66	a
Nite White 22% Excel	11	18.75	5.68	7.01-28.09	a
Opalescence PF 10%	12	17.41	3.01	13.13-23.42	a
Opalescence 20%	12	17.38	4.74	9.66-25.1	a
Rembrandt 15%	12	18.67	4.92	6.92-26.87	a
Nupro Gold 10%	12	18.59	3.94	12.98-27.08	а
Placebo	12	18.35	4.67	11.73-28.01	а

procedures were done following a random sequence. The specimens were stored in a humid environment at 37°C for 7 days.

In the shear bond strength tests, each specimen was positioned in a universal testing machine (Emic Ltd., São José dos Pinhais, PR, Brazil), with the enamel surface parallel to the machine's line of travel. A steel knife edge was placed over the specimen so that the shear force was directed at the bond surface. The specimens were loaded to fail at a crosshead speed of 0.5 mm/min.

The fractured surfaces of the specimens were examined visually with a stereomicroscope loupe at $\times 30$ (Kuraray, Tokyo, Japan) by two calibrated evaluators to classify the type of failure that occurred during the debonding procedure. Failure types were adhesive (adhesive failure), cohesive in enamel (dental substrate failure), cohesive in resin (resin failure), or mixed (cohesive in enamel and adhesive failure). The calibrated evaluators were blinded to the treatment category of the specimens. They evaluated the fracture modes in a random sequence. After reading the sample, the evaluators decided the final score for the fracture mode of the specimen. In case of disagreement, the evaluators determined the better score after a final discussion.

For the statistical analysis, the shear bond strength data were used to apply the one-way analysis of variance (ANOVA) with a significance level of p > .05. No statistical treatment was given to the analysis of the debonded samples, but a descriptive method was used.

RESULTS

The ANOVA did not show differences in shear bond strengths with the various treatment agents (p = .6896). Even results with the

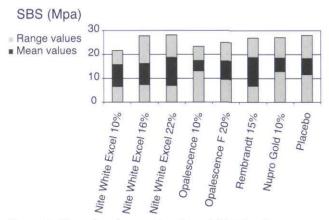


Figure 1. Shear bond strength values (MPa) for the treatment agents.

Group	No. of Failures (%)					
	Adhesive	Cohesive/Enamel	Cohesive/Resin	Mixed		
NiteWhite Excel 10%	9/12 (75.0)	1/12 (8.33)	0/12 (0)	2/12 (16.67		
Nite White Excel 16%	11/12 (91.67)	0/12 (0)	0/12 (0)	1/12 (8.33)		
Nite White Excel 22%	5/11 (45.45)	2/11 (18.18)	0/11 (0)	4/11 (36.36)		
Opalescence PF 10%	10/12 (83.33)	1/12 (8.33)	0/12 (0)	1/12 (8.33)		
Opalescence 20%	9/12 (75.0)	0/12 (0)	2/12 (16.67)	1/12 (8.33)		
Rembrandt 15%	7/12 (58.33)	0/12 (0)	2/12 (16.67)	3/12 (25.0)		
Nupro Gold 10%	10/12 (83.33)	1/12 (8.33)	0/12 (0)	1/12 (8.33)		
Placebo	11/12 (91.67)	1/12 (8.33)	0/12 (0)	0/12 (0)		

placebo agent (control group) did not differ from those of the other groups (Table 2 and Figure 1).

The examination of the debonded samples under a stereomicroscope loupe at $\times 30$ magnification showed that the fractures for all treatment agents were predominantly adhesive (Table 3). Resin failures were only observed for Opalescence 20% (2/12) and Rembrandt 15% (2/12). Mixed failures (resin and enamel) were not observed for the placebo agent, but a few were observed for Nite White 22% Excel (4/11) and Rembrandt 15% (3/12). Table 3 and Figure 2 show the results of fracture modes for all treatment agents evaluated.

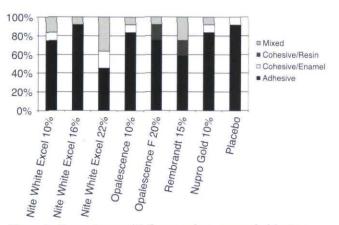


Figure 2. Percentages of failure modes as recorded by two evaluators for the treatment agents.

DISCUSSION

The demand for conservative esthetic dentistry has grown dramatically and so has the development of new nonrestorative treatments for discolored teeth. As nightguard vital bleaching has gained popularity as a conservative technique to lighten natural teeth, laboratories have rapidly introduced bleaching products into the market. Many of the newer systems contain carbamide peroxide in various concentrations, 2,3,30 with carbopol as a thickening agent to improve tissue adherence and allow for a time or sustained release of the whitening agent.³⁰ After the use of bleaching material for 2 to 6 weeks,¹ teeth are usually significantly lighter; preexisting restorations do not change and should be replaced to match the color of the teeth achieved with the bleaching treatment.5,6 One of the possible side effects of immediately restoring the teeth after the

completion of the treatment is the lower shear bond strength values.

This in vitro study evaluated the effects of different concentrations (10%, 15%, 16%, 20%, and 22%) of carbamide peroxide bleaching agents on enamel shear bond strength. Ten percent carbamide peroxide is the most frequently used concentration for at-home bleaching, as described by Haywood and Heymann,¹ and has American Dental Association acceptance.³¹ As 10% carbamide peroxide agents require a prolonged period of time to obtain significant whitening results, the use of increased percentages of carbamide peroxide has been advocated for faster results²; unfortunately, an increase in tooth sensitivity has became a compounding problem with these concentrations.30 In the presence of saliva, 10% carbamide peroxide releases 3% hydrogen peroxide (which penetrates enamel and dentin to lighten the tooth) and 7% urea.4 In products with higher concentrations of carbamide peroxide, higher concentrations of hydrogen peroxide are released, possibly providing a reduction of the adhesive system's shear bond strength.

In-office bleaching with hydrogen peroxide at the concentration of 35% and home-use bleaching with 10% carbamide peroxide seem to reduce the immediate postbleaching bond strength of resin to enamel.^{8–18} The lower shear bond strengths immediately after bleach-

ing procedures can be explained by physical and chemical alterations in enamel. Although the roughness of the surface and an increase in porosity caused by the loss of mineral content might eliminate the effects of a decrease in adhesiveness,^{7,9,24} it is suggested that the quality of the composite bond is compromised through a decreased number of resin tags, owing to the polymerization inhibition taking place and the relative amount of Ca on the enamel surface (which affects bonding).7-21,32 The inhibition is a result of bleaching agent use since dentin may act as an oxygen reservoir and the oxygen interferes with the curing of resin tags.7,10,12,22 The loss of minerals during bleaching procedures has also been suggested as a possible cause for a decrease in adhesiveness. 3,7,26,27,33-39

The delay of bonding restorations for 1 to 2 weeks is a common procedure to recover the normal bond strength values,7,12,15,16,22 even when 10% carbamide peroxide agents have been used.7,8,13,22,23 However, Josey and colleagues and Murchinson and colleagues found no differences in enamel bond strength values when evaluating 10% carbamide peroxide and using artificial saliva during or after the bleaching treatment.19,20 Nonetheless, a delay in bonding procedures should be considered to avoid the problems in enamel bonding procedures that generally follow a bleaching treatment.

In this study, shear bond strength tests were performed after the specimens were stored for 15 days in artificial saliva. This period might have reduced the possible interactions between the residual peroxides and the cure of the adhesive system and composite resin immediately after the completion of the bleaching treatment, and allowed for a recovery of the minerals from artificial saliva. In in vitro studies, the postbleaching period storage in artificial saliva for 15 days can also ensure an initial recovery of the shear bond strength to the higher values, as observed in a previous study.27 During and after the proposed bleaching treatments, samples were kept immersed in artificial saliva, which contains calcium and phosphate ions,28 which might have contributed to mineral gain and, consequently, raised the bond strength.

Our results indicated that there are no significant differences in shear bond strength among the different concentrations (10%, 15%, 16%, 20% and 22%) of carbamide peroxide agents. Even when a placebo agent was used, no differences were reported. It is possible that morphologic or structural changes in enamel owing to exposure to bleaching agents for 42 days were repaired by the storage in artificial saliva during the elapsed time between bleaching and the restorative procedure.27 McCracken and Haywood revealed a decrease in enamel microhardness related to the

treatment with a product containing carbopol.³⁴ The absence of carbamide peroxide on the placebo agent did not contribute to mineral content preservation.^{27,34} This performance may be due to the mineral loss caused by carbopol and glycerin during the treatment for 42 days, as related by Basting and colleagues.²⁷

Although significant changes in relative oxygen concentrations of enamel when using 10% carbamide peroxide were not shown by Perdigão and colleagues,7 the reduction in bond strengths has been attributed to unknown residual peroxide-derived substances at or near the enamel surface or in dentin and permeated in the interprismatic substance at the time of the exposure.7,12,22 Scanning electron microscope (SEM) examinations of enamel treated with carbamide peroxide revealed the presence of a granular, more-porous-appearing resin and evidence of bubbling that could be the result of oxidizing reactions owing to the entrapment of peroxide along interprismatic spaces.^{10,11} Once the entrapped peroxide is eliminated (during the time by which delaying bonding procedures are delayed), the enamel surface shows an increase in adhesiveness owing to the reduction in surface and subsurface contaminants, which, in turn, results in more effective etching and resin penetration.^{10,11,15} In the present study, all the specimens were kept in a remineralizing solution for

2 weeks prior to restorative procedures. The results revealed that artificial saliva contributed to mineral enhancement and therefore improved adhesive properties for the fragments bleached with different concentrations of carbamide peroxide agents or a placebo product.

Since shear bond strength is reduced by its own biomechanics,^{40,41} it is essential that a failure mode analysis be done to evaluate whether a fracture has occurred at the adhesive interface. The evaluation of the fracture modes showed mostly adhesive failures for all the agents evaluated. Although the correlation between the shear bond strength and failure patterns was not statistically evaluated, and the visual inspection with a light microscope of fractured sample surfaces may not have truly exposed the actual nature of the fractures as compared with an SEM evaluation,42 the fracture data show that lower and higher concentrations of carbamide peroxide bleaching agents can induce failures in the adhesive system and composite resin. Therefore, it appears that concentration of the carbamide peroxide does not affect failures. Zidan and colleagues showed that most resin-enamel fractures (mixed fractures) occur within the adhesive-resin composite complex, very close to the etched enamel surface, and can be misleading because of the intimate adaptation of this layer into the etched enamel.42 However, the fractures can be evaluated with light microscopy, which is inexpensive and easily performed.

The increasing quality of adhesive materials might also be a reason for the occurrence of dental cohesive failures. An ethanol and waterbased adhesive system was used in this experiment. Ethanol is known to reduce the surface water and increase enamel bond strength.^{18,21} This was confirmed in an experiment performed by Spyrides and colleagues demonstrating that the use of Single Bond-an ethanolbased adhesive-on dentin treated with a 10% carbamide peroxide agent did not seem to reverse the drop in shear bond strength values.²² The bonding values appear to be related to the presence of a remineralizing solution and to the period elapsed before the restorative treatment. Furthermore, the results show that all the agents subjected to shear bond forces provide preservation of the dental structure and the tags formed in the enamel. Therefore, the hypothesis that the residual peroxide and/or oxygen is a factor that affects the polymerization of the adhesive systems and composite resins may be taken into account.

CONCLUSION

It can be concluded that concentrations of carbamide peroxide agents varying from 10 to 22% cause no differences in shear bond strengths of enamel to an adhesive system after 15 days of storage in artificial saliva.

DISCLOSURE AND ACKNOWLEDGMENT

We are thankful for the financial support of Foundation for Research Support of São Paulo State—Grants 99/11735-2 and 98/14425-1.

The authors do not have any financial interest in the companies whose materials are discussed in this article.

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COMMENTARY

SHEAR BOND STRENGTH OF ENAMEL TREATED WITH SEVEN CARBAMIDE PEROXIDE BLEACHING AGENTS

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This study is one of a growing number of studies over the past decade that evaluate the effect of tooth bleaching products on enamel-composite bond strengths. It is a timely topic because of the widespread use and acceptance of this form of esthetic enhancement by patients, and the ever-increasing number of companies that introduce "new" products in every year. Since practitioners are often faced with the task of restoring teeth that have been first subjected to either home or in-office bleaching, it is crucial to understand the potential impact this may have on adhesive procedures and how to minimize or avoid it.

Current light-cured dental adhesives polymerize by a free radical polymerization that involves light-activated redox initiators. As pointed out by the authors, the problem with enamel that has been subjected to a bleaching regimen is that it collects hydrogen peroxide that has been released from the breakdown of carbamide peroxide. The low molecular weight of hydrogen peroxide allows it to penetrate completely through the enamel and into the dentin. The embedded hydrogen peroxide leaches out over time, decomposing into water and oxygen. This released oxygen causes incomplete polymerization of the adhesive resin that is in contact with these areas.^{1,2}

The authors distinguish their experimental approach from previous studies by choosing to evaluate various concentrations of currently available home bleaching products to observe their effects on enamel–resin composite bond strength. Their results confirm what the majority of previous studies in this area have shown, that a delay of 2 weeks after the cessation of bleaching allows enamel-to–resin composite bond values equal to those of nonbleached controls. The current authors did not study the effect of their bleaching products on immediate or 1-week bond strengths. The assumption the reader is asked to make is that there would be a significant reduction on shear bond values of enamel to resin composite because of the bleaching regimen. The

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