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Clinical Evaluation of 15% Carbamide Peroxide on the Surface Microhardness and Shear Bond Strength of Human Enamel

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Clinical Relevance

The clinical use of whitening chemicals should provide patients with some degree of certainty that there are no detrimental effects over time. Loss of surface hardness and polymerization inhibition are areas of concern in the literature and need clinical validation for both the practitioner and patient.

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SUMMARY

Purpose: This clinical evaluation compared a neutral sodium fluoridated whitening product to a neutral non-fluoridated whitening product in terms their effects on human enamel surface microhardness (SMH) and human enamel/resin composite shear bond strength (SBS) following various treatment times. Materials and Methods: Subjects were evaluated for enamel SMH and enamel/resin SBS following 15% carbamide peroxide (CP) with and without potassium nitrate and fluoride (PF). Twenty subjects (80 first or second premolars), who were treatment-planned for premolar extraction due to orthodontic therapy, were allocated into two groups, A and B. Group A received 15% CP, while Group B received 15% CP with PF. Each patient had a control tooth, a 14-day treatment + 14-day recovery tooth, a 14-day treatment + no recovery tooth and a 4-day + no recovery tooth. Each tooth was fur-

ther divided into two testing surfaces; the facial surface was used for SMH, while the lingual surface was used for SBS. Results: The results of this study determined that there was no statistically significant difference between the effects of the two products on SMH and enamel/resin SBS. Additionally, there was no statistically significant difference between the treatment specimens compared to the controls in terms of SMH. However, there was a significant difference between the treatment groups compared to the controls in terms of enamel/resin SBS. Conclusion: Within the limitations of this clinical study, 15% CP with and without PF does not seem to alter the SMH of human enamel. However, 15% CP with and without PF significantly reduced enamel/resin SBS immediately following tooth whitening therapy, up to 14 days post-treatment.

INTRODUCTION

Esthetic/cosmetic dentistry has become an increasingly popular topic in the United States, yet, surprisingly, this procedure dates back to the late 1800s.[1] Whitening chemicals for human enamel can be administered professionally by primary dental providers or they can be purchased over the counter by the consumer. Most whitening chemicals have been tested to some degree, and current data would suggest that professional "at-home" systems are the most efficacious. While whitening has become a well-accepted cosmetic treatment for teeth, the long-term clinical effects, such as changes in enamel surface microhardness (SMH) and enamel/resin shear bond strength (SBS), are not documented in the current available literature.

The American Dental Association's (ADA) accepted "at-home" vital whitening agent is 10% carbamide peroxide; however, most dentists' material of choice is carbamide peroxide in a 15% concentration.[2-4] The reason for the higher concentration utilization is not documented, but it is most likely due to product advertisement and trends. The tooth whitening process routinely involves placing a commercial whitening solution onto a mineralized enamel surface. However, there is no documentation as to whether minerals are released from the enamel structure, to what degree the release may occur and whether such release is clinically permanent.

Studies can be found in the literature on the adverse effects of 10-15% CP on the chemical and physical characteristics of enamel. These studies utilized several analytical methodologies, including SEM,[4] profilometry,[5] microhardness,[6] calcium loss[7] and infrared absorption spectroscopy.[8] Of all the analytical tests available,[9] microhardness testing has been the predominant choice for researchers to evaluate possible mineral loss due to whitening chemicals.[6-7,10-30] Many studies have found that CP solutions cause morphological changes to the surface of enamel.[11-16,18,22-23,25-29] However, other studies have found no significant alterations.[6,10,17,19-22,24] The question remains as to whether the possible changes that are found are transient, permanent or clinically significant.

Many manufacturers of whitening chemicals have begun to add various concentrations of fluoride to their whitening gels. With the current understanding of the enamel demineralization and remineralization cycle in the presence of fluoride products, it is not unreasonable to conclude that, if whitening products do not elicit surface demineralization, then fluoride benefits are minimal in terms of remineralization potential during vital whitening therapy.

There have been many reports regarding the relationship between bleaching agents and the bond strength of composite materials to enamel following bleaching. Many investigators have reported a severe decrease in the average bond strength of composites to bleached versus unbleached enamel.[31-32] Some researchers have reported that, only when composite was bonded immediately after bleaching, the bond strength was significantly reduced.[33] Other researchers have given examples of ways to counteract the adverse bleaching effects so that no statistical difference in bond strength was observed. Some examples of counteracting mechanisms include exposing the enamel specimens to artificial saliva, water or saline solution.[34] Additionally, it has been suggested that one remove the superficial enamel layer prior to bonding.[35]

Various theories have been proposed to explain why bond strength might affect bleached teeth. Changes in the enamel structure are one explanation; for example, a loss of mineral content and increased porosity manifesting as an over-etched appearance with a loss of the prismatic enamel form.[36] It has also been noted that the resin tags are reduced in number, are less defined and shorter in bleached enamel.[37] In addition, it has been proposed that, theoretically, the enamel pores, dentin and dentinal fluid could act as a peroxide/oxygen reservoir, resulting in oxygen concentrating on the surface of the enamel and preventing a complete cure of some resin tags.[32] In this last case, alcohol-based bonding agents may minimize oxygen's inhibitory effects through the interaction of alcohol with residual oxygen.

Consistent with the preceding oxygen inhibition theory, Sung and others[35] evaluated OptiBond, All-Bond 2 and One-Step for the shear bond strength of a hybrid composite to enamel treated with a 10% carbamide peroxide bleaching system. The groups using OptiBond demonstrated no statistical decrease in bond strength between the bleached and unbleached group. The All-Bond 2 and One-Step bonding agents demonstrated a

significant reduction in bond strength among the bleached specimens when compared to the unbleached controls. However, Lai and others[38] found a similar reduction in bond strengths of both acetone and alcohol-based resin adhesives following 10% carbamide peroxide bleaching to enamel.

Research has been conducted to overcome the oxygen inhibition effects by adding an anti-oxidative solution before resin adhesive bonding.[38-40] The *in vitro* results are promising in terms of using sodium ascorbate to reverse oxygen inhibition; however, these anti-oxidative solutions need to be tested clinically.

Table 1: *Population Demographics*

Group A 15% CP			Group B 15% CP/PF		
Subject	**Gen (Age)**	**Location**	**Subject**	**Gen (Age)**	**Location**
1	M (15)	Indianapolis	1	M (11)	Indianapolis
2	F (12)	Indianapolis	2	M (16)	Indianapolis
3	F (13)	Indianapolis	3	M (16)	Indianapolis
4	F (11)	Indianapolis	4	F (17)	Indianapolis
5	F (13)	Louisville	5	F (17)	Louisville
6	F (15)	Louisville	6	M (12)	Louisville
7	F (13)	Louisville	7	F (15)	Louisville
8	M (16)	Louisville	8	M (17)	Louisville
9	M (17)	Louisville	9	M (15)	Louisville
10	M (15)	Louisville	10	F (16)	Louisville
	Mean Age: (14)			Mean Age: (15)	
	4 Males 6 Females			6 Males 4 Females	

There have been many laboratory studies in the areas of surface microhardness and shear bond strength on enamel following vital whitening therapy; however, there was a need to evaluate these characteristics clinically. Therefore, this prospective clinical study provided clinical data for comparison against the extensive controversial laboratory data published in the literature.

Table 2: *Study Design and Treatment Groups*

Control (No Treatment)	LL= Lower Left
Treatment Days 1-14 + 14 Days Recovery (14tx + 14 r)	LR= Lower Right
Treatment Days 15-28 + No Recovery (14tx + nr)	UL= Upper Left
Treatment Days 25-28 + No recovery (4tx + nr)	UR= Upper Right

METHODS AND MATERIALS

Study Design and Allocation

In this clinical study, subjects were evaluated for enamel surface microhardness (SMH) and enamel/resin shear bond strength (SBS) following bleaching with 15% carbamide peroxide (CP) with and without potassium nitrate and fluoride (PF) (Opalescence Mint Flavored 15% CP and 15% CP/PF, Ultradent Products Inc, South Jordan, UT, USA). Twenty subjects (80 first or second premolars) were systematically allocated into two groups, A and B. Group A (10 patients) received 15% CP and Group B (10 patients) received 15% CP with PF. Systematic allocation was performed via every other patient starting with Group A. Study demographics are listed in Table 1. Each tooth was further divided into two testing surfaces; the facial surface was used for SMH and the lingual surface for SBS.

The inclusion criteria were the following: willingness to sign a consent form, the ability to make all four/five necessary appointments, good oral hygiene, all four premolars having been erupted into the oral cavity for at least one year or having at least 50% of the clinical crown erupted, being able to speak English and having reliable transportation to and from the clinic. The exclusion criterion were the following: restored premolars planned for extraction, pregnancy, gross oral pathology, high DMFT and demineralized areas, systemic diseases with oral ramifications and taking any medications that effect salivary flow and composition.

Subjects meeting the inclusion and exclusion criterion were identified by orthodontists in private practice and were referred for screening evaluation and qualification. The orthodontists provided no treatment to the subjects, only subject referral. Once the subjects were qualified, all proper paperwork was completed in accordance with Indiana University Institutional Review Board (IRB) 0511-13 regulation for human studies.

In Vivo Specimen Preparation

Once the subjects were identified, qualified and randomized, a flat surface was created with a 169L carbide bur (Brasseler USA, Savannah, GA, USA) onto the facial and lingual surfaces of all the premolars treatment planned for extraction. The flat surface was approximated towards the incisal natural flat contour, where the enamel is thickest on the crown. These areas were free of white spots, caries, stains and/or enamel fractures to the best of the primary investigators visu-

al ability under 3x Loop magnification. This reduction in enamel was limited to achieving a flattened area approximately 4 mm x 4 mm.

After the flattened surfaces were created, a resin composite polishing kit (Super-Snap Rainbow, Shofu Corp, San Marcus, CA, USA) was used from coarse to fine grit to smooth and polish the flattened enamel under light water irrigation.

Whitening Tray Fabrication

Two sets of alginate hydrocolloid impressions (Chromaclone, Ultradent Products Inc) were taken in disposable trays (COE Plastic Trays, GC America Inc, Alsip, IL, USA) following the disking procedure and poured with a vacuum mixed gypsum stone product (Microstone, Whip Mix Corp, Louisville, KY, USA) according to manufacturer's recommendations. One set of gypsum replica models was used to create the custom-made whitening trays (Sof-Tray 0.035", Ultradent Products Inc), and the other set was used for clear occlusal custom resin bonding stint fabrication.

Reservoirs for the whitening gel were placed on the facial and lingual surfaces of the gypsum replicas over the flattened surfaces of teeth 5(4)-12(13)-28(29) using LC Block-out Resin (Ultradent Products Inc), except for the lower left, #20 (21) control. The whitening trays and resin bonding stints were made with the help of an Ultra-Form (Ultradent Products Inc) vacuum former.

Resin Bonding Stint Design

Clear occlusal stints (Biocryl Material, 5 mm X 125 mm X 125 mm, Great Lakes Orthodontics, LTD, Tonawanda, NY, USA) were fabricated on the gypsum models around stainless steel (SS) copings (Metz Machine Shop) perpendicular to the flattened bonding areas. The stints were fabricated from the gypsum replica of each patient in the dry laboratory using the Ultra-Form (Ultradent Products Inc) vacuum former. The standardized SS copings were 1.2 mm internal diameter, 2.4 mm external diameter and 2.4 mm long. The SS copings were fastened perpendicular to all the flattened areas on the gypsum replica models with a tiny amount of cyanoacrylate (superglue). Once in place, the clear occlusal stint was formed around the fastened SS copings and along the dentition using a vacuum former and it was then sectioned. This resulted in two stints, with one SS coping in each for the maxillary arch and the same for the mandibular arch (four stints with one coping each). The stints were removed from the gypsum replicas with the SS coping inside and the residual cyanacrylate, if any, removed from the base of the coping.

Patient Take-Home Kit and Schedule

The custom mandibular whitening trays were delivered seven days from being created in the testing areas; the seven-day delay allowed for tray fabrication and all the flattened, polished areas to be exposed to the patients' saliva and dentifrice fluoride. The subjects did not receive any other in-office topical fluoride therapy following the disking and polishing procedures. During the study, the subjects were instructed to use an over-the-counter ADA-accepted dentifrice (1100 ppm) twice daily using a manual toothbrush.

The subjects' take-home packages included one syringe of whitening solution (Group A 15% CP; Group B 15% CP/PF), a mandibular tray and a tray case for treatment initiation. The mandibular tray was worn overnight (8 hours) on days 1-14, then discarded on day 15. At that time, a maxillary tray was delivered to the subjects with another syringe of whitening solution (Group A 15% CP; Group B 15% CP/PF) to wear overnight (8 hours) on days 15-28. The control and treatment specimens are listed in Table 2.

The subjects were also provided with a take-home laminated comprehensive schedule calendar, a demo color wheel gel applicator and a personal color wheel gel applicator that was color-coded to guide them through the whitening process and time changes. Figure 1 represents the treatment timeline for all subjects enrolled in the study. The schedule was thoroughly explained to both the subject and the legal guardian who signed the consent form. Additionally, gel application and tray maintenance was physically demonstrated. The subject and guardian were then asked to repeat the demon-

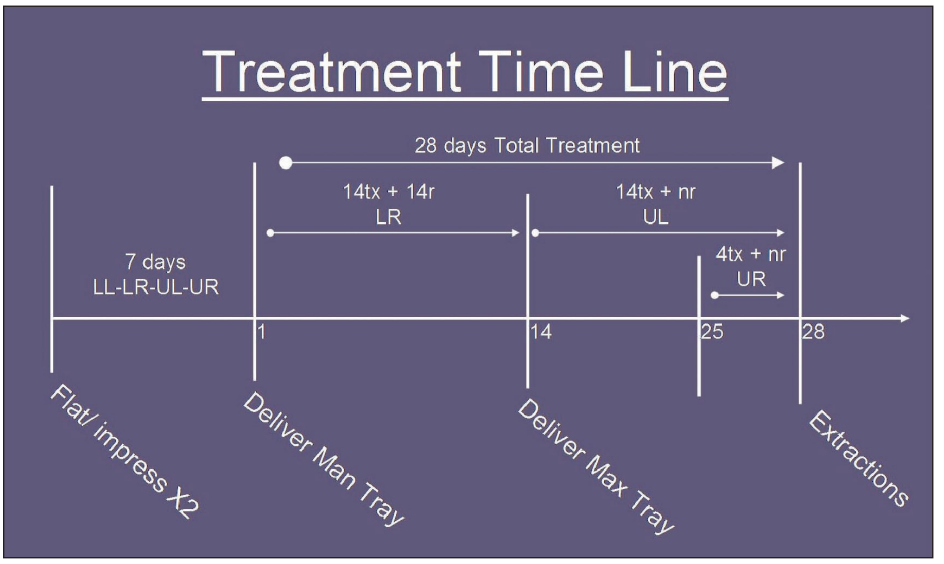

Figure 1. *Treatment timeline.*

stration to ensure complete understanding of the application process.

Between three and five subjects were started on a single appointment, and the schedule was staggered for time management among several orthodontic and oral surgery offices. Each subject was compensated upon successful completion of the study in accordance with IRB 0511-13.

Specimen Removal and Resin Bonding

On day 29, all four teeth from each patient were atraumatically extracted by an oral surgeon using elevators, and the extraneous tissue was removed. The specimens were wiped clean with 2 x 2 gauze to prepare them for bonding. The lingual flattened bonding areas were conditioned with 35% phosphoric acid (Ultra-Etch, Ultradent Products Inc) for 20 seconds, rinsed with deionized water and air-dried until frosty white in appearance. An ethanol-based resin adhesive (Optibond Solo Plus Unidose, Kerr USA, Orange, CA, USA) was then placed and light cured for 30 seconds on continuous mode (Optilux 501, Kerr USA). The entire extracted specimen was then oriented into the stint, and, on each specimen, resin composite (Kerr .4 Vita A1, Kerr USA) was placed down the coping in two increments and light cured for 60 seconds each on continuous mode (Optilux 501, Kerr USA). Once all four specimens were bonded, the clear occlusal stints were gently cut away with a new #12 scalpel, leaving behind the SS copings with the bonded resin composite inside for shear bond strength testing (Figure 2). Prior to each patient's extraction, the quart tungsten halogen (QTH) light source was checked using a built in radiometer to ensure a digital output reading of at least 470 nanometers.

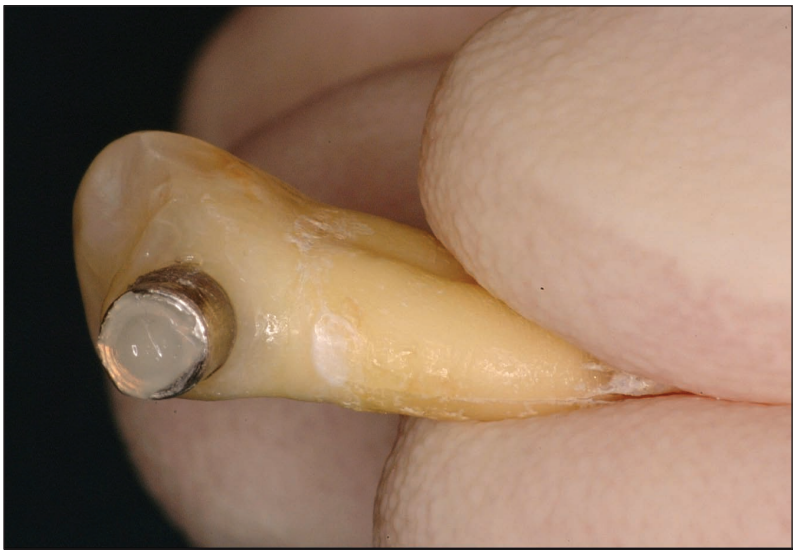

Figure 2. *Photograph of resin bonded extracted specimen.*

The bonded specimens were then placed in a 0.1% thymol solution as a transport media and stored at 4°C overnight until sectioned for analysis. Each subject had four labeled biohazard containers: C for control, LR for lower right, UR for upper right and UL for upper left.

Specimen Sectioning and Mounting

The whole specimens were sectioned in the mesial-distal direction and the root/roots removed using a water-cooled trimmer (Lapcraft L'il Trimmer, Lapcraft, Inc, Powell, OH, USA). The facial specimens were adjusted on the section line so that the cut or mounting surface paralleled the flattened testing surface. Four facial specimens at a time were placed on a flat acrylic slab, with the flattened testing area against the slab. The specimens were placed on an acrylic slab with sticky wax, and the cut surfaces were ground on a polishing machine and measured with a digital micrometer to create plano-parallel surfaces to the testing surfaces.

The lingual specimens were fastened into a stainless steel jig (Metz Machine Shop) four at-a-time, and the portion of specimen outside the jig was used for mounting in a Bencor Multi-T testing ring (Danville Engineering, San Ramon, CA, USA). A polymethymethacrylate material (Bosworth Fast-Tray, Bosworth Company, Skokie, IL, USA) was poured into the testing rings to secure the specimens in the testing ring.

Specimen Testing

Enamel SMH was measured using a Wilson 2100 Knoop Hardness Tester (Wilson Instruments, Norwood, MA, USA). Each facial enamel specimen was secured on a 1-inch square acrylic block with sticky wax and placed on the microhardness tester. Five indentations were placed in a diamond pattern around the center of the testing area using a Knoop diamond bur under a 50-g load for 15 seconds. The KHN was determined by measuring the length of the indentations (µm) using an image analysis system at 200x magnification (Wilson-Wolpert Image Analysis Software, version 3.5.0, Pentium 3 computer interfaced with the Wilson 2100 Hardness Tester).

Once polymerized, the lingual specimens were removed from the perpendicular mounting jig and placed in the testing machine so that the shear blade was 90° to the coping and mounting ring. The shear blade was oriented next to the specimen/coping interface to minimize any cantilever forces and optimize evaluating the shear bond strength throughout the collective bonding area.

Enamel/resin SBS was evaluated using the Bencor Multi-T stainless steel system (Danville Engineering) in combination with the MTS Universal Testing Machine (MTS Systems Corp, Cary, NC, USA). A vertical load was applied to the piston with the shear blade on the mounted specimen coping until specimen-resin adhesive failure. The vertical load crosshead speed was applied to the piston by using the MTS Universal Testing Machine and MTS TestWorks 4 software (version 4.07a) at a rate of 0.5 mm/minute, with the failure load being recorded in megapascals (MPa).

Data Analysis

For SMH, the five Knoop indentation sites from each specimen were averaged to determine surface microhardness for that specimen. A two-sample *t*-test was performed to compare the control groups for the two products. Analysis of variance (ANOVA) was performed to compare the treatment groups against the controls and for comparisons of the treatment groups. ANOVA included a random effect to correlate the four teeth within each subject. In addition, the SMH for the treated teeth were divided by the SMH for the control tooth for each subject in order to calculate the percentage reduction. ANOVA was performed to compare the treatment groups for differences in percentage reduction in SMH, again with a random effect to correlate the teeth within each subject.

For SBS, a two-sample *t*-test was performed to compare the control groups for the two products. Analysis of variance (ANOVA) was performed to compare the treatment groups against the controls and for comparisons of the treatment groups. ANOVA included a random effect to correlate the four teeth within each subject.

Sample Sizing

A pilot study was conducted to obtain enough information for sample size calculations. Twelve premolars from three patients (ages 12-18; two males, one female) received no treatment products; however, these premolars had flattened areas created on the facial surfaces and were extracted after 35 days (7 + 28). The results indicated the mean control enamel surface microhardness was approximately 350, with a between-subject standard deviation of 11 and a within-subject correlation of 0.67. Therefore, for sample size calculations, the authors conservatively used a within-subject correlation of 0.5 and a between-subject standard deviation of 15 and 10% of the control mean (35) as a clinically significant difference in means. Assuming an overall 5% significance level for the comparisons, with a sample size of 10 subjects each for the non-fluoridated and fluoridated groups, the study had 90% power to detect differences of 35 between any two treatments within each group and 85% power to detect differences of 35 between the non-fluoridated and fluoridated groups for any treatment.

Between-subject standard deviation of shear bond strength is estimated to be 5.6 megapascals (MPa). From the surface microhardness pilot study in conjunction with this study, a within-subject correlation of 0.5 was accepted. Assuming a 5% significance level for each comparison, with a sample size of 10 subjects each for the non-fluoridated and fluoridated groups, the study had 80% power to detect differences of 5.6 between any two treatments within each group and 80% power to detect differences of 7.5 between the non-fluoridated and fluoridated groups for any treatment.

Study Materials

All corresponding materials used in the study were from the same lot numbers and expirations dates. All treatment products were from the same lot numbers in order to minimize possible manufacturer variances in active ingredients or processing conditions.

RESULTS

SMH data by product and treatment are graphically represented in Figure 3. The mean values show that both products and all treatments had similar Knoop SMH values and standard deviation to the controls. The controls for this study had similar Knoop SMH values and standard deviations compared to the pilot study.

Knoop SMH values, divided by the control values by product and treatment, resulted in no decrease in SMH compared to the control. Comparisons between product controls and between products per se determined that the controls did not have significantly different surface microhardness values (p=0.9996), and there were no significant surface microhardness differences between products overall (p=0.8856) or for any treatment (p=0.9962) (p=0.9889) (p=0.9994). Comparing the treatment surface microhardness values against the control, it was determined that none of the treatments had significantly different Knoop SMH values from the control. Comparisons between treatments determined that 14-day treatment + 14 days of recovery, 14-day treatment + no recovery and 4-day treatment + no recovery did not have significantly different Knoop SMH values. Comparisons between products divided by control determined that there were no significant Knoop SMH differences between products overall or for any treatment. A comparison between treatments divided by the control values determined that all treatments did not differ significantly from the control.

SBS data by product and treatment are shown in Figure 4. Both products exhibited similar SBS values for the control and for the various treatments provided. Graphically, the treatments of 14 days + 14 days

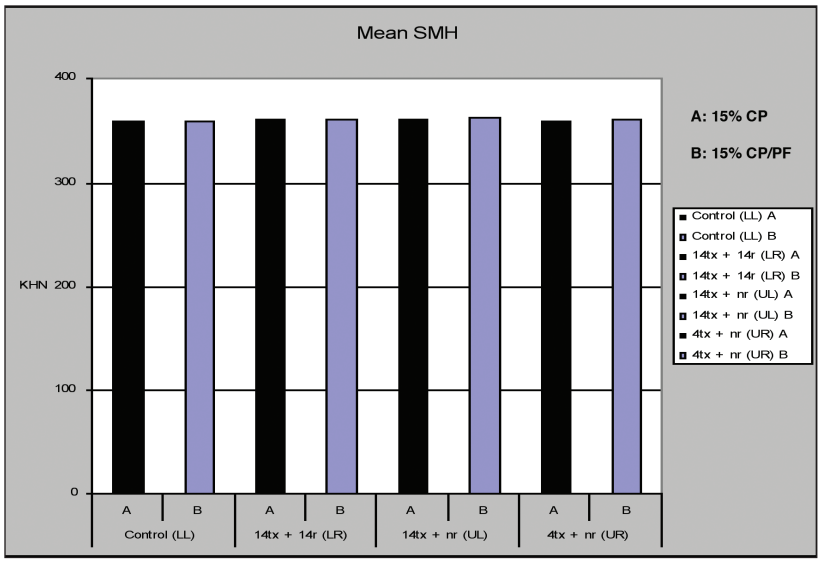

Figure 3. *Mean Knoop (KHN) SMH values for Groups A and B.*

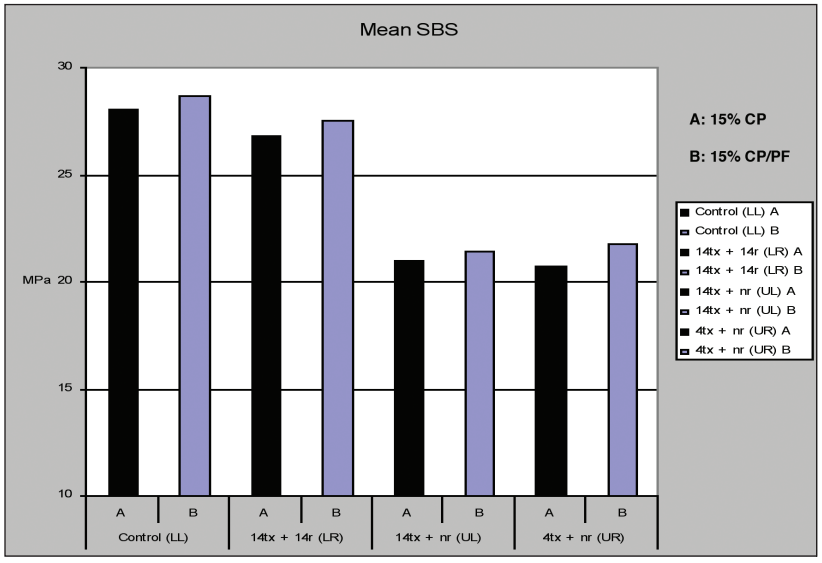

Figure 4. *Mean SBS values for Groups A and B.*

recovery, 14 days + no recovery and 4 days + no recovery were lower in shear bond strength compared to the control values. Additionally, the treatments of 14 days + no recovery and 4 days + no recovery showed a similar decrease in shear bond strength for both products.

SBS divided by the control determined that the 15% CP treatment group of 14 days + 14 days recovery reached 95.7% of the control value. Additionally, the treatment group using 15% carbamide peroxide for 14 days + no recovery and 4 days + no recovery reached 75% and 73.8% of the control value, respectively. The 15% CP/PF treatment group using the product for 14 days + 14 days recovery reached 96.1% of the control value. Additionally, the treatment group of 14 days + no recovery and 4 days + no recovery reached 74.4% and 75.5% of the control value, respectively. Each treatment for both Groups A and B exhibited similar loss of SBS as a percent of the control values. Comparisons between product controls determined that the controls did not have significantly different SBS values (p=0.9889). Comparisons between products determined that there were no significant SBS differences between products overall (p=0.6465) or for any treatment (p=0.9983) (p=0.9814) (p=0.9460). Comparing the treatment SBS values against the control, it was determined that all treatments had significantly lower shear bond strength than the controls. Comparisons between treatments determined that the 14-day treatment + 14 days of recovery had significantly higher SBS than the 14-day treatment + no recovery and 4-day treatment + no recovery, but the 14-day treatment + no recovery and 4-day treatment + no recovery did not have significantly different SBS. Comparisons between products divided by the control determined that there were no significant SBS differences between products overall or for any treatment. Comparisons between treatments divided by control values determined that 14-day treatment + 14 days of recovery had significantly higher SBS than 14-day treatment + no recovery and 4-day treatment + no recovery, but the 14-day treatment + no recovery and 4-day treatment + no recovery did not have significantly different SBS.

DISCUSSION

The results of this clinical study on enamel surface microhardness (SMH) determined that there was no significant difference between the two products or treatment regimens compared to the control baseline. These results are consistent with *in situ* studies that found no statistically significant loss of surface enamel hardness.[22,24,30] Additionally, the current understanding of fluoride incorporation into the enamel crystal is that it needs saturation conditions that favor fluoride incorporation, as well as the presence of available nucleation sites for the new minerals. In this study, the surface microhardness was not altered by the whitening solutions; therefore, the fluoridated group did not benefit from the fluoride ion in the solution in terms of ion exchange that might have resulted in an increased microhardness over baseline. Also, both products used in this study were at neutral pH, ranging from 6.5 to 7.5. If the fluoridated product was at a more acidic pH, then the fluoride benefits might have potentially caused a more protective effect following surface enam-

el demineralization and remineralization, forming a more acid-resistant fluoride-rich crystal. Basting and others[13] and Attin and others[18] showed that, if enamel specimens are demineralized prior to fluoridated product application, then the driving force will allow incorporation of the fluoride ion for increased surface microhardness, although baseline values may not be reached. Intuitively, treatment with neutral sodium fluoride whitening products on non-demineralized surfaces, such as in the current study, would offer no benefit.

Clinical relevance must include the salivary potential for remineralization and appropriate representation of the oral environment. Many *in vitro* study models are limited by design but have tried to include this salivary potential for remineralization and test microhardness on highly polished specimens.[11,16,19,23,25-26,30] Several *in situ* studies have included the potential remineralizing effects of human saliva, which were excluded in some previous *in vitro* studies.[13,22,24,30]

The results of this clinical study on enamel/resin composite shear bond strength (SBS) determined that there was no statistically significant difference between the control values or treatments provided by these two products. However, all treatments were significantly lower than the control values for both groups. These results are consistent with many *in vitro* studies that have found lower shear bond strengths immediately following whitening therapy.[33,38-45] Additionally, like other research,[39,40,42,45-46] at least 14 days of recovery following vital whitening therapy is needed to regain optimal bonding characteristics to approach control values. However, one can question whether there is a clinically significant difference in SBS. For example, is there a clinical difference between 28.1 ±3.8 MPa and 20.8 ± 3.4 MPa in terms of clinical success or longevity? The answer to this question is that it depends on the clinical situation in which the adhesive bonding treatment is planned. It may be less clinically significant for endodontic access closure following non-vital whitening compared to diastema closures on a patient with end-to-end anterior occlusion who home-whitened for 14 days. Additionally, knowing that waiting the 14 days will yield better adhesion via longer resin tags, there is no question as what to do for patients.

The treatment of 14 days + no recovery and 4 days + no recovery did not differ significantly between products. However, the treatments were significantly lower in SBS than the control values. Understanding that, if patients do a "touch-up" whitening for four days immediately prior to cosmetic bonding, they will experience similarly poor adhesive properties as that of a patient whitening for the first time for 14 days, will improve the clinical longevity of resin bonding in dental practices.

The mechanism by which resin adhesive bonding is affected by whitening chemicals is still controversial. What is understood is that there is a mechanism and clinical failures to support these theories. Obviously, only the ramifications of the mechanisms and not the possible mechanisms, themselves, were evaluated in this clinical study. The most common and theorized mechanism of adhesive interference is the oxygen inhibition theory. Oxygen-free radicals from the degradation of hydrogen peroxide transverses the enamel crystal and into the dentin tubules. This oxygen inhibition decreases the ability of the ethanol-based resin adhesive to penetrate enamel for the micromechanical retention needed for clinical success. In agreement with this study, there have been several *in vitro* studies that have recommend waiting at least two weeks before adhesive bonding with an ethanol-based adhesive to allow the oxygen-free radicals to dissipate.[39-40,42,45-46] There has been some recommendation in the literature to treat the whitened surfaces with anti-oxidizing chemicals to eliminate the oxygen-free radicals if immediate bonding is inevitable.[38-40] The success with this concept has mixed results and no long-term clinical data to support its efficacy. In conflict with this study, Sung and others[35] determined no significant difference with bleached versus unbleached enamel using an ethanol-based bonding adhesive *in vitro*. However, it was shown that an acetone-based bonding adhesive was significantly affected when comparing bleached versus unbleached enamel *in vitro*. Similar to this study, Lai and others[38] showed a significant decrease with both an acetone and ethanol-based adhesive immediately following bleaching *in vitro*.

CONCLUSIONS

The clinical results of this study need further comparisons to other resin adhesive bonding agents and bonding techniques immediately following whitening therapy on a wider age range of individuals.

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References

1. Haywood VB (1992) History, safety, and effectiveness of current bleaching techniques and applications of the Nightguard vital bleaching technique *Quintessence International* **23(7)** 471-488.
2. Haywood VB & Heymann HO (1989) Nightguard vital bleaching *Quintessence International* **20** 173-176.
3. Kelleher MGD & Roe FJ (1999) The safety in use of 10% carbamide peroxide for bleaching teeth under the supervision of a dentist *British Dental Journal* **187** 190-194.

4. Haywood VB, Heymann HO, Leech T, Crumpler D & Bruggers K (1990) Nightguard vital bleaching: Effects on enamel surface texture and diffusion *Quintessence International* **21(10)** 801-804.
5. McGuckin RS, Babin JF & Meyer BJ (1992) Alterations in human enamel surface morphology following vital bleaching *Journal of Prosthetic Dentistry* **68(5)** 754-760.
6. Seghi RR & Denry I (1992) Effects of external bleaching on indentation and abrasion characteristics of human enamel *in vitro* demineralization *Journal of Dental Research* **71(6)** 1340-1344.
7. Haywood VB & McCracken MS (1996) Demineralizing effects of 10 percent carbamide peroxide *Journal of Dentistry* **24(6)** 395-398.
8. Oltu Ü & Gürgan S (2000) Effects of three concentrations of carbamide peroxide on the structure of enamel *Journal of Oral Rehabilitation* **27(4)** 332-340.
9. Arends J & Bosch JJ (1992) Demineralizing and remineralizing evaluation techniques *Journal of Dentistry* **71** 924-928.
10. Joiner A & Cooper Y (2004) Evaluation of a 6% carbamide peroxide tooth whitening gel on enamel and dentin microhardness *in vitro Journal of Dentistry* **32** 27-34.
11. de Oliveria R, Basting RT, Rodrigues AL, Rodrigues JA & Serra MC (2003) Effects of a carbamide peroxide agent and desensitizing dentifrice on enamel microhardness *American Journal of Dentistry* **16(1)** 42-46.
12. Potocnik I, Kosec L & Gaspersic D (2000) Effect of 10% carbamide peroxide bleaching gel on enamel microhardness, microstructure, and mineral content *Journal of Endodontics* **26(4)** 203-206.
13. Basting RT, Rodrigues Junior AL & Serra MC (2001) The effect of 10% carbamide peroxide bleaching material on microhardness of sound and demineralized enamel and dentin *in situ Operative Dentistry* **26(6)** 531-539.
14. Akal N, Over H, Olmez A & Bodur H (2001) Effects of carbamide peroxide containing bleaching agents on the morphology and subsurface hardness of enamel *Journal of Clinical Pediatric Dentistry* **25(4)** 293-296.
15. Bitter NC (1998) A scanning electron microscope study of the long-term effect of bleaching agents on the enamel surface *in vivo General Dentistry* **46(1)** 84-88.
16. Pinto CF, de Oliveira R, Cavalli V & Giannini M (2004) Peroxide bleaching agent effects on enamel surface microhardness, roughness and morphology *Pesquisa Odontologica Brasileira* **18(4)** 306-311.
17. Pugh G Jr, Zaidel L, Lin N, Stranick M & Bagley D (2005) High levels of hydrogen peroxide in overnight tooth-whitening formulas: Effects on enamel and pulp *Journal of Esthetic and Restorative Dentistry* **17(1)** 40-45; discussion 46-47.
18. Attin T, Vollmer D, Wiegand A, Attin R & Betke H (2005) Subsurface microhardness of enamel and dentin after different external bleaching procedures *American Journal of Dentistry* **18(1)** 8-12.
19. Teixeira EC, Ritter AV, Thompson JY, Leonard RH Jr & Swift EJ Jr (2004) Effect of tray-based and trayless tooth whitening systems on microhardness of enamel surface and subsurface *American Journal of Dentistry* **17(6)** 433-436.
20. Lewinstein I, Fuhrer N, Churaru N & Cardash H (2004) Effect of different peroxide bleaching regimens and subsequent fluoridation on the hardness of human enamel and dentin *Journal of Prosthetic Dentistry* **92(4)** 337-342.
21. Ünlü N, Çobankara FK, AltinÖz C & Özer F (2004) Effect of home bleaching agents on the microhardness of human enamel and dentin *Journal of Oral Rehabilitation* **31(1)** 57-61.
22. Justino LM, Tames DR & Demarco FF (2004) *In situ* and *in vitro* effects of bleaching with carbamide peroxide on human enamel *Operative Dentistry* **29(2)** 219-225.
23. Basting RT, Rodrigues AL Jr & Serra MC (2003) The effects of seven carbamide peroxide bleaching agents on enamel microhardness over time *Journal of the American Dental Association* **134(10)** 1335-1342.
24. Araujo EM, Baratieri LN, Vieira LC & Ritter AV (2003) *In situ* effect of 10% carbamide peroxide on microhardness of human enamel: Function of time *Journal of Esthetic and Restorative Dentistry* **15(3)** 166-173; discussion 174.
25. Rodrigues JA, Basting RT, Serra MC & Rodrigues AL (2001) Effects of 10% carbamide peroxide bleaching materials on enamel microhardness *American Journal of Dentistry* **14(2)** 67-71.
26. Attin T, Kielbassa AM, Schwanenberg M & Hellwig E (1997) Effect of fluoride treatment on remineralization of bleached enamel *Journal of Oral Rehabilitation* **24(4)** 282-286.
27. ten Cate JM & Arends J (1977) Remineralization of artificial enamel lesions *in vitro Caries Research* **11** 277-286.
28. Featherstone JD, Cutress TW, Rodgers BE & Dennison PJ (1982) Remineralization of artificial caries-like lesions *in vivo* by a self-administered mouth rinse or paste *Caries Research* **16(3)** 235-242.
29. White DJ & Featherstone JD (1987) A longitudinal microhardness analysis of fluoride dentifrice effects on lesion progression *in vitro Caries Research* **21(6)** 502-512.
30. Shannon H, Spencer P, Gross K & Tira D (1993) Characterization of enamel exposed to 10% carbamide peroxide bleaching agents *Quintessence International* **24(1)** 39-44.
31. Ben-Amar A, Liberman R, Gorfil C & Bernstein Y (1995) Effect of mouthguard bleaching on enamel surface *American Journal of Dentistry* **8** 29-32.
32. Titley KC, Torneck CD, Ruse ND & Krmec D (1993) Adhesion of a resin composite to bleached and unbleached human enamel *Journal of Endodontics* **19(3)** 112-115.
33. Dishman MV, Covey DA & Baughan LW (1994) The effects of peroxide bleaching on composite to enamel bond strength Dental Materials **10(1)** 33-36.
34. Swift EJ & Perdigão J (1998) Effects of bleaching on teeth and restorations *Compendium* **19(8)** 815-820.
35. Sung EC, Chan SM, Mito R & Caputo AA (1999) Effect of carbamide peroxide bleaching on the shear bond strength of composite to dental bonding agent enhanced enamel *The Journal of Prosthetic Dentistry* **82(5)** 595-599.
36. Josey AL, Meyers IA, Romaniuk K & Symons AL (1996) The effect of vital bleaching technique on enamel surface morphology and the bonding of composite resin to enamel *Journal of Oral Rehabilitation* **23(4)** 244-250.

37. Titley KC, Torneck CD, Smith DC, Chernecky R & Adibpar A (1991) Scanning electron microscopy observations on the penetration and structure of resin tags in bleached and unbleached bovine enamel *Journal of Endodontics* **17(2)** 72-75.

38. Lai SC, Tay FR & Cheung GS (2002) Reversal of compromised bonding in bleached enamel *Journal of Dental Research* **81(7)** 477-481.

39. Bulut H, Türkün M & Kaya AD (2006) Effect of an antioxidizing agent on the shear bond strength of brackets bonded to bleached human enamel *American Journal Orthodontics & Dentofacial Orthopedics* **129(2)** 266-272.

40. Türkün M & Kaya AD (2004) Effect of 10% sodium ascorbate on the shear bond strength of composite resin to bleached bovine enamel *Journal of Oral Rehabilitation* **31(12)** 1184-1191.

41. Cavalli V, de Carvalho RM & Giannini M (2005) Influence of carbamide peroxide-based bleaching agents on the bond strength of resin-enamel/dentin interfaces *Pesquisa Odontologica Brasileira* **19(1)** 23-29.

42. Cavalli V, Reis AF, Giannini M & Ambrosano GM (2001) The effect of elapsed time following bleaching on enamel bond strength of resin composite *Operative Dentistry* **26(6)** 597-602.

43. García-Godoy F, Dodge WW, Donohue M & O'Quinn JA (1993) Composite resin bond strength after enamel bleaching *Operative Dentistry* **18(4)** 144-147.

44. Nour El-din AK, Miller BH, Griggs JA & Wakefield C (2006) Immediate bonding to bleached enamel *Operative Dentistry* **31(1)** 106-114.

45. Bishara SE, Oonsombat C, Soliman MM, Ajlouni R & Laffoon JF (2005) The effect of tooth bleaching on the shear bond strength of orthodontic brackets *American Journal of Orthodontics & Dentofacial Orthopedics* **128(6)** 755-760.

46. Basting RT, Rodrigues JA, Serra MC & Pimenta LA (2004) Shear bond strength of enamel treated with seven carbamide peroxide bleaching agents *Journal of Esthetic and Restorative Dentistry* **16(4)** 250-259.